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PRACTICAL METHODS

FOR THE

Iron and Steel Works Chemist

BY

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NEW CASTLE, PA.

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PART I

THE LABORATORY

LOCATION

The iron and steel works laboratory should be located at a point where you can secure good drainage and lighting, and at the same time be as far as possible from the noise, dirt, dust and vibration of the plant. However, none of the above should be allowed to interfere with locating at a point where the samples can be received and analyses reported in the shortest possible time.

PLAN

The plan of the laboratory building should be controlled largely by the size of the plant, and consequently the amount of work to be done. However, the larger plants should have a building not over 35 feet wide, so as to give good light from each side, and long enough to accommodate the work to be done. It should be of one story, at least 15 feet high, and a basement at least eight feet high. The latter should be one-half above ground, so as to be easy of access, and also for good lighting.

The laboratory proper should be divided into four rooms only; a general work room at one end and of sufficient size to accommodate all the routine analytical work. Next to it should be the balance room. Each of these two rooms should occupy the full width of the building. The other end of the building, or that portion on opposite side of balance room from general work room, may be divided into two rooms, one for an office and the other one as special work room.

The basement should be divided into the following apartments: One for preparing and filing iron and steel samples, one for preparing samples of ore, coke, coal, limestone, etc.; one for a fan for ventilating and heating building; one for storing acids, glassware, etc.; and a lavatory. There should be an outside entrance to basement and a stairway leading from the iron and steel sample room to the general work room.

CONSTRUCTION

The building should be as near fireproof as it is possible to make it. A good grade of brick is the most suitable material, and should have good concrete foundation. The inner walls of the laboratory proper should be of enamelled brick and the floor a good grade of asphalt. The basement should be finished with cement.

GENERAL INFORMATION

Have laboratory fitted with ample hood room and arranged with cupboards with locks, for each chemist. Have sink and water supply at middle of each hood so that it can be used by a person working on each side. If good water pressure, use it for an individual suction for each chemist and the exhaust water foi washing up. Give each man glassware, etc., to be used by himself only. Mark all glassware with "Diamond Ink" when you give it out, so that each man can keep his own. Use one size only of funnels for general work and large enough for a 11 cm. filter paper. Two sizes of filter papers are enough for general work, a nine cm. and 11 cm., with a few 18 cm. for special purposes. For this latter you will need a few four-inch funnels. Two qualities of paper are enough, a "double washed" ashless, and the other of good quality and unwashed. For the ashless, the author uses B. & A. "double washed," "A" thickness, which are low enough in ash for all practical purposes in iron and steel work and are also very cheap. For the other quality I have never been able to find anything so good as Munktel's No. 1 F. They are a thin rapid filter and retain precipitates well. They are also very low in ash and not expensive.

The argand burners give best results for most purposes. Have a few bunsen burners and blast-lamps, and these can best be made by your pipe fitter and fitted to your gas and air pipes, so as to do away with all rubber hose connections. A number of these have been in use in our laboratory for several years with no signs of giving out, and they also give a better heat than other kinds. Two muffles, of double walls so as not to heat laboratory, one heated with bunsen burners for burning cokes and other purposes when a high temperature is not needed; the other should be heated by a blast so as to get a higher temperature for rapid ignition and high temperature work. These muffles, properly built in your hood by a good brick layer, are as good as any you can get, and in many ways are better. In addition to this, you should have one or more steam and hot plates, all of which should be placed in hoods. In burning in platinum use a muffle wherever it is practicable, as a direct flame is more or less injurious to the platinum. An oven heated by electric light globes is a very convenient method of drying or heating at temperatures below 130° C. Build your oven any length you need to accommodate the work, and about seven inches deep and nine inches high, with a door covering the entire front side and hinged at bottom. A perforated shelf should extend the entire length, with space enough below for the light globes. These should be placed closely together the entire length of oven and inserted from the back of oven so that the glass bulbs only are inside. The heat can be regulated by turning on as many lights as is necessary. Line inside with asbestos board.

For handling the steel drillings in the routine work small tin boxes are best. These should have numbers stamped on the lids and there should be several sets for as many days' steel. These boxes can be used over again and again and are more convenient than paper containers. Paper containers are best for iron drillings and also for steel in open hearth work where it is necessary to mark same otherwise than simply with a number. Cokes, limestones, slags, etc., are kept in paper containers; however, a slag sample so kept is not reliable only for a very short time. Ore samples which are generally kept for a considerable length of time are best kept in bottles with cork stoppers. All routine analysis of iron and steel and furnace slags should be recorded in a book for that purpose. All other samples on entering the laboratory should be given a number and entered in a general analysis book, together with date of receipt, description of sample and any other information. This book should have a place opposite for recording the analysis and also a place for date of reporting same. The laboratory should have a small library of the best up-to-date books on all analyses pertaining to the iron and steel industry; also the best works on the theory and practice of iron and steel making.

For drilling iron and steel samples, a horizontal drilling machine is much better than the upright. It should be fitted with a chuck for holding a one-half-inch drill and an attachment with rack and pinion for holding sample. The rack and pinion is best operated by a four-arm lever. Use high speed steel for drills. For preparing irons which are too hard to drill, have a block of tool steel with hole two inches in diameter and about three inches deep, fitted with plunger for using sledge to pound up sample. For holding this, a block set in the ground and on top of this a casting about four inches thick with a hole cast in same for the tool steel block to set in is best.

For preparing ores, cokes, limestone, etc., a Gates crusher and grinder and bucking-boards of chrome steel are best, with also a small crusher of some other good make for crushing small samples. Use forced draft system of ventilating, and then by keeping windows and doors closed you compel all air and fumes to pass out through the hoods. By passing air through a box heated by steam pipes the same system can be used for heating. Also by taking the air supply from a cool source or an underground tunnel you can keep the laboratory cool even during the hottest weather. Taken from the same source during the winter months it will not require so much heating.

The matter of furniture, fitting and apparatus, as well as the arrangements for the working of each individual chemist, cannot receive too careful attention, for these things have much to do with the proper working of a laboratory when so large a volume of work must be gotten out in so short a space of time.

Make your methods as simple as possible. Do away with all complicated apparatus except for special work.

In glassware use as few kinds and sizes as possible and insist upon your supply house giving you uniform goods and just what you specify.

MISCELLANEOUS NOTES

Have but one dilution of HNO_3 (sp. gr. 1.20), and if a more dilute acid is needed dilute with H_2O at time of using.

HCl is also kept in but one dilution, one part acid to one part H_2O by volume. This is used for washing and in dissolving irons and steels for sulfur in evolution method. However, for many purposes where a dilute HCl is used it is just as good practice to add the H_2O and HCl separately instead of keeping a reagent bottle of 1 : 1 HCl.

Porcelain dishes are less soluble than most glassware, and for this reason are better to use for many determinations.

About 98 per cent. of the SiO_2 is removed by taking fusions and soluble silicates to dryness once. The balance is obtained by evaporating the filtrate after removing the SiO_2 from first evaporation. In material high in SiO_2 this will be an appreciable amount. The amount of SiO_2 in first filtrate is largely determined by the temperature at which it has been dehydrated, the length of time exposed to same, and also the length of time the SiO_2 is exposed to the acid solution before filtering. In many cases where it is desirable to remove the SiO_2, as in phosphorus determinations, it may be done by adding HF and evaporating until all the HF is expelled, thereby shortening the method materially.

On account of the solubility of glassware, especially in alkaline solutions, in all cases where it is practicable, it is best to use platinum or porcelain dishes. In taking solutions to dryness for removal of SiO_2, it is best to expel the last of the acid by placing in an oven heated to about 120° C for one-half to one hour. The same should be placed in oven while it is still moist with the acid.

STANDARDS

Every iron and steel laboratory should have a full line of reliable standards. I have found of use the following:

Steel, for color carbons and for checking up combustion carbons, of about the following percentages: 0.09, 0.14, 0.20, 0.35, 0.55, 0.75, 0.95. The steel of lower carbon percentages can also be used for manganese, sulfur and phosphorus.

An iron, for silicon, sulfur, phosphorus, manganese, graphite and total carbon.

An ore, for iron, manganese, phosphorus, silica, alumina, lime and magnesia.

A coke, for volatile, sulfur, phosphorus and ash.

A coal, for volatile, sulfur, phosphorus and ash.

A limestone, for phosphorus, sulfur, silica, alumina, iron, lime, magnesia and sulfur.

A slag, for silica, alumina, lime, magnesia and sulfur.

A ferro-manganese, for manganese, silicon and phosphorus.

The Elements Used in This Work

Element	Symbol	Valence	Atomic Weight	Melting Point, °C	Boiling Point, °C	Specific Gravity	Specific Heat
Aluminum	Al	3	27.1	657.3		2.67	0.2143
Antimony	Sb	-ous 3					
		-ic 5	120.2	630.5	1500-1700	6.70	0.0508
Arsenic	As	-ous 3					
		-ic 5	75.0	Red Heat	449.5 Subs.	5.63	0.0814
Barium	Ba	Generally 2	137.4	850	950	4.00	0.0470
Bismuth	Bi	-ous 3					
		-ic 5	208.0	269	1435	7.67-9.93	0.0380
Boron	B	3	11.0	Infusible		2.68	0.3660
Bromin	Br	1, 3, 5	79.96	—7.3	63.05	3.15	0.0843
Cadmium	Cd	2	112.4	321.7	778	8.45	0.0567
Calcium	Ca	Generally 2	40.1	780	Non-Volatile	1.58	0.1670
Carbon	C	2, 4	12.0	Infusible	3600 Subs.	2.33-2.52	0.4590
Chlorin	Cl	1, 3, 5	35.45	—102	—33.6	1.38 (liq.)	0.1800
Chromium	Cr	-ous 2					
		-ic 3	52.1	1515		7.01	0.1000
Cobalt	Co	-ous 2					
		-ic 3	59.0	1530		8.957	0.1070
Copper	Cu	-ous 1					
		-ic 2	63.6	1065	2100	8.952	0.0950
Fluorin	F	1	19.0	—223	—187		0.2600
Gold	Au	-ous 1					
		-ic 3	197.2	1065		19.50	0.0324
Hydrogen	H	1	1.008	—256.5	—252.5	0.0692-(Air 1.0)	2.3000
Iodin	I	1, 3, 5	126.97	114	184.35	4.94	0.0541
Iron	Fe	-ous 2					
		-ic 3	55.9	1550-1600		7.79	0.1138
Lead	Pb	2, 4	206.9	327	1450-1600	11.35	0.0306
Magnesium	Mg	2	24.36	632.6	1100	1.70	0.2499
Manganese	Mn	-ous 2					
		-ic 3	55.0	1245		8.03	0.1217
Mercury	Hg	-ous 1					
		-ic 2	200.0	—38.85	357	13.60	0.0319
Molybdenum	Mo	2, 3, 4, 6	96.0			8.56	0.0722
Nickel	Ni	-ous 2					
		-ic 3	58.7	1484		9.50	0.1082
Nitrogen	N	3, 5	14.01	—210.5	—196.18	0.971-(Air 1.0)	0.3600
Oxygen	O	2	16.0	—230	—184	1.105-(Air 1.0)	0.2500
Phosphorus	P	3, 5	31.0	44.2	287.3	1.84	0.1895
Platinum	Pt	-ous 2					
		-ic 4	194.8	1780		21.15	0.0324
Potassium	K	1	39.15	62.5	757.5	0.86	0.1655
Silicon	Si	4	28.4	1200		2.49	0.2030
Silver	Ag	1	107.9	955		10.53	0.0560
Sodium	Na	1	23.05	97.6	877.5	0.98	0.2934
Strontium	Sr	Generally 2	87.6	900		2.542	0.1740
Sulfur	S	2, 4, 6	32.06	115-119	444.6	2.07	0.1776
Tin	Sn	-ous 2					
		-ic 4	119.0	232	1450-1600	7.20	0.0562
Tintanium	Ti	3, 4	48.1	3000		3.588	0.1300
Tungsten	W		184.0	1700		18.30	0.0334
Vanadium	V		51.2	1680		5.50	
Zinc	Zn	2	65.4	419	918	7.37	0.0955

Table of Factors

Found	Sought	Factor	Found	Sought	Factor
2Fe	Fe_2O_3	1.4293	$BaCO_3$	C	0.06074
Fe	FeO	1.2862	$BaCO_3$	CO_2	0.2229
3Fe	Fe_3O_4	1.3816	$BaCO_3$	CO	0.1409
Fe_2O_3	2Fe	0.6996	$BaCO_3$	CH_4	0.0812
Fe_2O_3	2FeO	0.8999	$BaCO_3$	C_nH_{2n}	0.0710
$3Fe_2O_3$	$2Fe_3O_4$	0.9666	CO_2	C	0.2727
FeO	Fe	0.7775	CO_2	$CaCO_3$	2.2750
2FeO	Fe_2O_3	1.1182	CO_2	$MgCO_3$	1.9173
3FeO	Fe_3O_4	1.0749	SiO_2	Si	0.4702
Fe_3O_4	3Fe	0.7281	Cl	NaCl	1.6502
Fe_3O_4	3FeO	0.9266	Cl	KCl	2.1044
$2Fe_3O_4$	$3Fe_2O_3$	1.0345	2Cl	CaCl	1.5656
Mn	MnO	1.2909	2Cl	MgCl	1.3436
2Mn	Mn_2O_3	1.4364	AgCl	Cl	0.2466
3Mn	Mn_3O_4	1.3879	K_2O	2KCl	1.5821
Mn	MnO_2	1.6000	K_2O	K_2SO_4	1.8490
Mn_2O_3	2Mn	0.6962	KCl	Cl	0.4752
Mn_3O_4	3Mn	0.7205	2KCl	K_2SO_4	1.1686
Mn_3O_4	3MnO	0.9301	K_2SO_4	SO_3	0.4592
MnO_2	Mn	0.6250	K_2PtCl_6	2KCl	0.3071
$Mn_2P_2O_7$	2Mn	0.3873	K_2PtCl_6	K_2O	0.1941
Al_2O_3	2Al	0.5303	Na_2SO_4	Na_2CO_3	0.7463
$AlPO_4$	Al	0.2220	Na_2SO_4	SO_3	0.5631
$2AlPO_4$	Al_2O_3	0.4185	Na_2O	Na_2CO_3	1.7085
CaO	$CaCO_3$	1.7843	Na_2O	Na_2SO_4	2.2731
CaO	$CaSO_4$	2.4275	Na_2O	2NaCl	1.8840
CaO	CaF_2	1.3916	2NaCl	Na_2SO_4	1.2150
CaO	$CaCl_2$	1.9608	2NaCl	SO_3	0.6843
$CaCO_3$	CaO	0.5604	Cu_2S	2Cu	0.7986
$CaSO_4$	CaO	0.4119	CuO	Cu	0.7990
$CaSO_4$	$CaCO_3$	0.7352	$PbSO_4$	Pb	0.6829
MgO	$MgCO_3$	2.0902	$PbCrO_4$	Pb	0.6406
MgO	$MgCl_2$	2.3602	NiO	Ni	0.7858
MgO	$MgSO_4$	2.9836	SnO_2	Sn	0.7881
$MgCO_3$	$MgSO_4$	1.4274	Sb_2O_3	2Sb	0.8336
$Mg_2P_2O_7$	P_2O_5	0.6376	Sb_2O_5	2Sb	0.5703
$Mg_2P_2O_7$	2P	0.2784	Sb_2O_4	Sb	0.7898
$Mg_2P_2O_7$	2MgO	0.3629	Sb_2S_3	2Sb	0.7145
$Mg_2P_2O_7$	$2MgCO_3$	0.7575	$Zn_2P_2O_7$	2Zn	0.4291
$Mg_2P_2O_7$	$2MgSO_4$	1.0813	Bi_2O_3	2Bi	0.8966
SO_3	K_2SO_4	2.1778	Cr_2O_3	2Cr	0.6846
SO_3	$CaSO_4$	1.7007	2Cr	Cr_2O_3	1.4607
SO_3	CaO	0.7007	TiO_2	Ti	0.5880
SO_3	$MgSO_4$	1.4904	TiO_2	$TiO_2P_2O_5$	2.7728
SO_3	Na_2SO_4	1.7755	WO_3	W	0.7931
SO_3	$FeSO_4$	1.8981	WO_3	$PbWO_4$	1.965
$BaSO_4$	S	0.1373	WO_3	Na_2WO_4	1.2677
$BaSO_4$	SO_3	0.3429	$PbWO_4$	W	0.4045
$BaSO_4$	Ba	0.5885	$PbWO_4$	WO_3	0.5100
$BaSO_4$	BaO	0.6571	$(Pb_2V_2O_7)_2PbO$	4V	0.1384
$BaSO_4$	C	0.5140	$(Pb_2V_2O_7)_2PbO$	$2V_2O_5$	0.2773
$BaSO_4$	CO_2	0.1885	V_2O_5	2V	0.5625

Table of Molecular Formulae and Molecular Weights

Name	Molecular Formulae	Molecular Weight
Ferrous Oxid	FeO	21.9
Ferric "	Fe_2O_3	159.8
Ferric " (Magnetic)	Fe_3O_4	231.7
Manganous Oxid	MnO	71.0
Manganic "	Mn_2O_3	158.0
Trimanganese Tetroxid	Mn_3O_4	229.0
Manganese Peroxid	MnO_2	88.0
Manganese Pyrophosphate	$Mn_2P_2O_7$	284.0
Aluminum Oxid (Alumina)	Al_2O_3	102.2
Aluminum Phosphate	$AlPO_4$	122.1
Calcium Oxid (Lime)	CaO	56.1
Calcium Carbonate	$CaCO_3$	100.1
Calcium Sulfate	$CaSO_4$	136.16
Calcium Fluorid	CaF_2	78.1
Calcium Chlorid	$CaCl_2$	111.0
Magnesium Oxid (Magnesia)	MgO	40.36
Magnesium Carbonate	$MgCO_3$	84.36
Magnesium Sulfate	$MgSO_4$	120.42
Magnesium Chlorid	$MgCl_2$	95.26
Magnesium Pyrophosphate	$Mg_2P_2O_7$	222.72
Phosphorus Pentoxid	P_2O_5	142.0
Potassium Sulfate	K_2SO_4	174.36
Sodium Sulfate	Na_2SO_4	142.16
Ferrous Sulfate	$FeSO_4$	151.96
Sulphur Trioxid	SO_3	80.06
Barium Oxid	BaO	153.4
Barium Carbonate	$BaCO_3$	197.4
Barium Sulfate	$BaSO_4$	233.46
Carbon Dioxid	CO_2	44.0
Carbon Monoxid	CO	28.0
Methane	CH_4	16.032
Illuminants	C_nH_{2n}	$C_2H_4 = 28.032$
Potassium Chlorid	KCl	74.6
Sodium Chlorid	$NaCl$	58.5
Silver Chlorid	$AgCl$	143.35
Potassium Oxid	K_2O	94.3
Potassium-Platinic-Chlorid	K_2PtCl_6	485.8
Sodium Peroxid	Na_2O_2	78.1
Sodium Carbonate	Na_2CO_3	106.1
Copper Oxid	CuO	79.6
Copper Sulfid	Cu_2S	159.26
Lead Sulfate	$PbSO_4$	302.96
Lead Chromate	$PbCrO_4$	323.0
Nickel Oxid	NiO	74.7
Tin Oxid	SnO_2	151.0
Antimonous Oxid	Sb_2O_3	288.4
Antimonic Oxid	Sb_2O_5	320.4
Diantimony Tetroxid	Sb_2O_4	304.4
Antimonous Sulfid	Sb_2S_3	336.58

Information to Aid in Rapid Calculations

1 Gram = 15.43 Grains
1 Meter = 39.37 Inches
1 Liter = 33.81 Fluid Ounces
1 Fluid Ounce = 29.57 Cubic Centimeters
1 Ounce (Avoirdupois) = 28.35 Grams
1 " Troy = 31.10 "
1 Grain = 64 Milligrams
1 U. S. Gallon = 231 Cubic Inches
1 Liter = 61.02 " "
1 Pound (Avoirdupois) = 7,000 Grains (453.59 Grams)
1 U. S. Gallon Water at 4° C = 58,353 Grains (3787.61 grams)

A column of water 44 mm. (1.732 inches) high is equal to 1 ounce pressure.

To convert parts per million into grains per gallon, multiply by 0.058353.

1 cu. ft. water = 62½ pounds.

STANDARD SOLUTIONS AND REAGENTS

STANDARD POTASSIUM BICHROMATE

8.85 grams $K_2Cr_2O_7$ to 1000 cc. of water.

Let stand several weeks and then standardize with a standard iron ore so that one cc. will equal one per cent. iron when using one gram sample.

STANNOUS CHLORID

A 30 per cent. solution of $SnCl_2$ in HCl (sp. gr. 1.10). As this solution rapidly changes to $SnCl_4$, it is best to make it up in small quantities and keep it in a cool, dark place.

MERCURIC CHLORID

A saturated solution of $HgCl_2$ in water.

POTASSIUM FERRICYANID (INDICATOR)

Ten mg. K_3FeCy_6 in 100 cc. water. Must be made up as used.

AMMONIUM PHOSPHATE

A 10 per cent. solution of $(NH_4)_2HPO_4$ in water. Make up in small quantities and never use a solution that has stood more than one month.

SODIUM THIOSULFATE

A 20 per cent. solution of $Na_2S_2O_3$ in water. This is not very stable and is best made up frequently.

ACETIC ACID

Use the 50 per cent. $HC_2H_3O_2$.

STANDARD POTASSIUM PERMANGANATE

6.5 grams $KMnO_4$ to 1000 cc. water. Let stand several weeks before standardizing. In making a more dilute solution for low manganese determination, add 150 cc. of the above solution to 1000 cc. water, and standardize after standing some time.

POTASSIUM PERMANGANATE (FOR OXIDATION)

Use 20 grams $KMnO_4$ to 1000 cc. of water.

MOLYBDIC SOLUTION

$$(a)\ \left.\begin{array}{l} MoO_3,\ 225\ \text{grams} \\ H_2O,\ 500\ \text{cc.} \\ NH_4OH,\ 500\ \text{cc.} \end{array}\right\}\ \begin{array}{l} \text{Mix and add a few} \\ \text{drops of ammonium} \\ \text{phosphate solution.} \end{array}$$

$$(b)\ \left.\begin{array}{ll} HNO_3,\ (1.42) & 950\ \text{cc.} \\ H_2O, & 1550\ \text{cc.} \end{array}\right\}\ \text{Mix.}$$

Pour (a) into (b), shake, let stand in a warm place 24 hours, filter and use.

SILICON MIXTURE

H_2O, 1275 cc.
HNO_3, 475 cc.
H_2SO_4, 350 cc.

Add the HNO_3 to the H_2O, then while blowing air through the mixture add the H_2SO_4 very slowly.

BROMIN WATER

A saturated solution of Br in water.

BARIUM CHLORID

A 10 per cent. solution of $BaCl_2$ in water.

AMMONIUM OXALATE

A saturated water solution of $(NH_4)_2C_2O_4$.

COPPER POTASSIUM CHLORID

Four hundred grams $(KCl)_2CuCl_2$ in 1000 cc. water. Let stand two weeks, filter through asbestos and add 75 cc. HCl.

ABSORBENT FOR EVOLUTION SULFUR METHOD

Two pounds Babbitt's lye and 1000 cc. NH_4OH added to one carboy of water. While blowing air through the above, add 12 grams starch which has been boiled up with 2000 cc. of water. Draw off into bottles for use.

STARCH SOLUTION FOR SULFUR IN SLAGS

Two grams starch boiled with 500 cc. water and to which is added five grams $ZnCl_2$. For use in doing Cu, etc., by the iodin method, this solution must be made fresh and without the $ZnCl_2$

STANDARD IODIN

I, 8.8 to 9 grams.
KI, 20 grams.

Dissolve in about 50 cc. water and make up to 3500 cc. with water. Let stand for one week and then standardize with standard iron or steel so that one cc. will equal 0.01 per cent. sulfur when using three grams sample. Keep from the light.

AMMONIUM PERSULFATE

One hundred and forty grams $(NH_4)_2S_2O_8$ in 1000 cc. water. Keep stock bottle in cool, dark place and this solution will be reliable for at least two weeks.

NITRIC ACID PLUS SILVER NITRATE

Five grams $AgNO_3$ in 1000 cc. H_2O. To this is added 2000 cc. HNO_3 (1.20). Keep stock bottle in the dark and use an amber colored reagent bottle.

STANDARD ARSENITE

As_2O_3, 20 grams.
Na_2CO_3, 60 grams.
H_2O, 200 cc.

Mix and boil until reaction is complete. Then filter and add water to make 2000 cc. Add about 120 cc. of the above solution to 2000 cc. of water for the standard solution. Standardize with a standard iron, steel or ore so that one cc. will equal 0.1 per cent. manganese when using 0.2 gram sample.

SOLUTIONS FOR TITRATION OF AMMONIOPHOSPHO-MOLYBDATE

Make a stock solution of NaOH by dissolving 157 grams in just enough water to not quite completely dissolve; let any carbonate settle, pour off the clear solution and add water to make 2000 cc. For standard solution use about 200 cc. of stock and 800 cc. water. Make a standard HNO_3 solution to balance by adding about 35 cc. to 3500 of water. Standardize with a standard iron, steel or ore so that one cc. will equal 0.01 per cent. phosphorus on two grams sample.

PHENOLPHTHALEIN (INDICATOR)

Two grams of the powder in 600 cc. alcohol and 400 cc. water. Add the NaOH solution, used in phosphorus work, drop by drop, until you get a pink color, then the HNO_3 until it is just discharged.

MAGNESIA MIXTURE

$MgSO_4$, 50 grams.
NH_4Cl, 50 grams.
H_2O, 400 cc.

Mix and add 200 cc. NH_4OH. Let stand several weeks and filter for use.

ESCHKA'S MIXTURE

Buy your MgO and Na_2CO_3 already mixed. Test for sulfur before using.

FUMING SULFURIC ACID ($H_2S_2O_8$)

Buy through your supply house.
One cc. absorbs eight cc. ethene.

CUPROUS CHLORID (ACID)

Place one-half inch of powdered or scale CuO in 2000 cc. bottle, also several heavy copper wires reaching from bottom to top of bottle. Fill with HCl (1.10) and shake frequently. When clear it is ready for use and can be poured into another bottle containing copper wire. Fill up bottle again with HCl. In this way a supply can always be kept on hand. One cc. absorbs at least one cc. CO.

POTASSIUM HYDRATE

Five hundred grams KOH in 1000 cc. water. Let settle and use only the clear solution. One cc. absorbs 40 cc. CO_2.

POTASSIUM PYROGALLATE

Five grams pyrogallic acid dissolved in 100 cc. of the KOH solution used for CO_2.

Note: When doing gases containing over 25 per cent. O, use a pyrogallate made with a KOH solution containing 120 grams KOH to 100 cc. water.

SODIUM CHLORID

Two grams NaCl (c. p.) to 1000 cc. water. For use in doing manganese by persulfate method.

BARIUM HYDRATE

Twenty grams $Ba(OH)_2$ to 1000 cc. H_2O.

AMMONIUM ACETATE

Twenty per cent. solution of $NH_4C_2H_3O_2$ in water.

PART II

IRON ORES

SAMPLING

The proper sampling is an easy matter, providing the ore is of a uniform degree of fineness. However, when there is fine and lump together, it presents many difficulties.

I have known a bessemer ore of eight per cent. SiO_2 to vary as much as three per cent. in SiO_2 between the lump and the fine, and the phosphorus would also vary as much as 0.012 per cent.

In sampling from a boat, the sample should be taken at several times during the unloading, and total sample should weigh from 500 to 2000 pounds. When taken from cars at least 10 pounds should be taken from each car, and this should be taken from at least six different parts of the carload. Make the sampling as nearly mechanical as possible.

PREPARATION OF SAMPLE FOR ANALYSIS

The sample should be well mixed and quartered and one-fourth of the whole amount placed in a large steam plate to dry for preparation of sample for analysis. About 25 pounds (10,000 grams) should also be taken at once and weighed and dried in an oven at 100° C until thoroughly dried, and weighed again for moisture.

In case of an ore containing many lumps, it is best to throw on a one-half-inch screen and crush all that will not pass through, and then mix all well together before quartering and setting to dry.

When dry, run through a crusher until all will pass a one-quarter-inch screen, then mix well and quarter to about 25 pounds. This should be run through a small crusher or grinder, adjusted so that the ore will pass a No. 8 sieve. This should again be well mixed and about two pounds run through the grinder, adjusted so that it will practically all pass a No. 80 sieve. This final sample should be well mixed and a portion dried at 100° C, bottled and stoppered while hot, and when cool is ready for analysis.

METHOD FOR IRON

Weigh out one gram sample in duplicate, moisten with five cc.

$SnCl_2$ solution, and add 20 cc. HCl, set on steam plate at near the boiling point for about two hours.

While hot, add $SnCl_2$, drop by drop, until the color has all disappeared, then add a few drops excess, cool by diluting well with cold water, add 10 cc. $HgCl_2$, stir well and titrate with standard solution of $K_2Cr_2O_7$, test a drop now and then on a white slab with a solution of K_3FeCy_6 until no blue coloration appears inside of one-half minute.

Note: Do not titrate in direct sunlight, as it acts on the K_3FeCy_6 and will give a blue color after all iron is oxidized.

Ores in which the iron is not all soluble in HCl, the residue should be fused and fusion added to the HCl solution before titration.

Ores containing organic matter should be ignited before dissolving in HCl.

METHOD FOR MANGANESE

One gram sample moistened well with water, add 25 cc. HCl and five cc. H_2SO_4, boil slowly until fumes of SO_3 come off freely, cool, add 50 cc. H_2O, boil, dilute to 100 cc., mix well and draw off 20 cc. with pipette, add to this 20 cc. of the HNO_3 plus $AgNO_3$ used in determining manganese in iron or steel, heat to boiling, remove from light and add 15 cc. $(NH_4)_2S_2O_8$ solution. Let stand several minutes, cool, add seven cc. NaCl solution and titrate with standard arsenite.

Note: The arsenite solution should be standardized by an ore in which the manganese has been determined gravimetrically.

For ores containing more than one per cent. Mn, use more of the $AgNO_3$ and $(NH_4)_2S_2O_8$.

Ores above two per cent. manganese should be done by the gravimetric method. See page 32.

If ores contain insoluble silicates of Mn, fuse residue, dissolve in dilute H_2SO_4 and add to soluble portion.

METHOD OF PHOSPHORUS

To five grams sample add 35 cc. HCl, boil slowly until all iron is in solution, dilute with water and filter, fuse residue with Na_2CO_3 and dissolve with water and HCl in separate beaker, take both to dryness, now add 25 cc. HCl (five to fusion and 20 to the other), evaporate until nearly to dryness and add 15 cc. HNO_3

(five to fusion and 10 to other), heat until reaction is over, then dilute fusion beaker with enough water to dissolve all salts, boil it and then pour enough of it into the other beaker so the acid will not cut the filter paper. Now filter into flask, pouring that portion containing the most iron through first, then wash fusion into other beaker and finally wash all on to the filter.

By the above procedure the solution can be kept small enough for precipitation of phosphorus without evaporating again.

Now add 15 cc. NH_4OH, shake up and add HNO_3, five cc. in excess of clearing solution. Heat or cool to 80° C and add 50 cc. molybdic, shake and let stand until settled, filter on suction, wash once with two per cent. HNO_3 and then well with water, return paper with precipitate to flask, add 50 cc. water free from CO_2 and enough of the standard NaOH to more than dissolve, stopper flask and shake well, wash off stopper into flask, add a few drops phenolphthalein and titrate with the HNO_3 until pink color just disappears. The difference between the number of cc. NaOH and the HNO_3 is the phosphorus. If solutions are standardized for two grams iron or steel, multiply results by 0.4.

Note: The final solution after adding molybdic should not be larger than 180 cc.

If ores contain small amounts of arsenic, precipitate so that final temperature will not exceed 40° C and do not let stand more than one-half hour.

If small amounts of titanium are present, fuse the final silicious residue with Na_2CO_3, dissolve in water, filter, make acid with HNO_3 and take to dryness, take up in small amount of HNO_3, filter and add to first filtrate before precipitating phosphorus.

The SiO_2 from the phosphorus determination after washing may be ignited and weighed for the SiO_2.

METHOD FOR SILICA, ALUMINA, LIME AND MAGNESIA

Moisten two grams sample well with water, add 25 cc. HCl and boil slowly until iron is dissolved, dilute and filter, fuse residue with seven times its weight of Na_2CO_3, dissolve in water in separate beaker, make acid with HCl and take both to dryness. Take up in 25 cc. HCl, adding part to each beaker, dilute with water, boil and filter, washing well with HCl (1.1) and hot water. Ignite residue and weigh as SiO_2. Use precautions for TiO_2 and $BaSO_4$.

ALUMINA

To the filtrate from the SiO_2 add one gram $(NH_4)_2S_2O_8$, heat to near boiling, make alkaline with NH_4OH and boil a few minutes to precipitate all Fe, Al and Mn. Filter and reserve filtrate for Ca and Mg. Add the filter containing the precipitate to a beaker containing 25 cc. HCl (1.10), and digest until all precipitate is dissolved, filter and dilute to 500 cc. in graduated flask, mix and divide into two equal portions by using a 250 cc. graduated flask. Transfer to large beaker, dilute to 350 cc., and finish in duplicate as follows: Add 30 cc. of 10 per cent. solution of $(NH_4)_2HPO_4$. Now add NH_4OH until you get a slight permanent precipitate. Now add one and one-half cc. HCl, which should clear the solution. Next add 50 cc. of a 20 per cent. solution of NaS_2O_3, bring to the boiling point and add at once 15 cc. $HC_2H_3O_2$ (50 per cent.), and 15 cc. $NH_4C_2H_3O_2$ (20 per cent. solution). Boil 10 minutes, filter, wash well with hot H_2O, burn in porcelain crucible and weigh as $AlPO_4$. Calculate to Al_2O_3.

LIME

To the reserved filtrate for lime and magnesia, add five cc. $(NH_4)_2C_2O_4$ solution and 10 cc. NH_4OH and evaporate to 150 cc., now add five cc. more NH_4OH and boil, filter off any CaC_2O_4, ignite and weigh as CaO.

Note: The lime will almost invariably contain Fe_2O_3 and Al_2O_3. This will not be precipitated if solution is made acid with HCl and heated after adding the $(NH_4)_2C_2O_4$, prior to adding the NH_4OH.

MAGNESIA

Cool the filtrate from the lime, make acid with HCl, add five cc. $(NH_4)_2HPO_4$ (10 per cent. solution), then while stirring add 25 cc. NH_4OH, stir frequently and let stand several hours, filter and wash well with water containing one-fourth its volume of NH_4OH, burn in porcelain crucible until white and weigh as $Mg_2P_2O_7$. Calculate to MgO.

SULFUR

Fuse one gram with 10 grams Na_2CO_3 and one-half gram KNO_3, dissolve in hot water and filter, wash well with hot water. Make acid with HCl and take to dryness, add three cc. HCl and 100 cc. water, boil, filter, heat to boiling and add slowly 10 cc.

$BaCl_2$ solution, boil for some time, let stand several hours, filter and wash well with hot water, ignite over argand or bunsen and weigh as $BaSO_4$. Calculate to S.

Note: If ore is low in silica the taking to dryness may be omitted; just acidulate with HCl and precipitate with $BaCl_2$.

If water solution of fusion will not filter, acidulate all with HCl and take to dryness. Take up in HCl, evaporate off most of the HCl, dilute with water, filter and precipitate as $BaSO_4$.

Na_2O_2 may be used instead of Na_2CO_3 and KNO_3 in fusing, but must be done in nickel crucible.

SIEVE TESTS

Use brass wire sieves 18 inches in diameter with sheet iron cover and copper pan for bottom sieve. Must be made in nest and of the following number of meshes to the linear inch: 2, 8, 20, 40, 60, 80 and 100.

Sample about 500 pounds ore, mix well and quarter to about 125 pounds (enough to make 100 pounds dry ore). After drying, divide into four about equal portions, and sieve each well in nest by rocking back and forth on floor. Then remove each sieve separately, beginning at top, and sieve well on paper, rapping side of sieve continuously until practically no more will pass through. Whatever gathers on the paper to be added to the next finer sieve.

The sum of all the weights remaining on the different sieves and in the pan to be used as the divisor in calculating the percentages of weights of ore remaining on each sieve and in the pan.

COKE AND COAL

SAMPLING

In sampling coke and coal, as in ores, the nearer mechanical you can make the operation the better. Take large and small pieces just as they are without breaking, being careful to take an average sample and not select the best pieces altogether, neither those containing an excess of foreign matter. A box about 18 inches square and 10 inches deep and fastened to a handle, is a good arrangement for catching coke or coal when running from the bottom of the car. This can be held under the car several times during the unloading and all dumped in one pile, where it can be thoroughly mixed and a smaller portion taken as representative of the carload. This is an especially good procedure in taking samples for moisture.

PREPARATION OF SAMPLE FOR ANALYSIS

If you wish to determine moisture, the sample should be at once crushed to about one-half-inch size, well mixed and quartered and at least 10 pounds weighed, and dried in oven at 100° C. From the loss in weight you can calculate the percentage of moisture. This sample can now be used to prepare sample for analysis. Crush and grind until all will pass a No. 8 sieve, mix well and pass a small portion through grinder, so adjusted that the sample will practically all pass a No. 60 sieve, mix well again and take a small sample for analysis.

Note: In drying a coal, it will be found to lose weight almost indefinitely, which is due to the loss of volatile matter. The sample should be dried from one-half to one hour, according to the amount of free water, and weighed. Then weighed again at the end of every 15 minutes until there is no appreciable loss in weight.

METHOD FOR VOLATILE MATTER

One gram is placed in a 30 cc. platinum crucible with a well fitting lid and all weighed. It is then heated for three and one-half minutes over a bunsen burner and then (without cooling) for three and one-half minutes over a blast-lamp. The loss in weight is the volatile matter.

Note: In order to make results as nearly comparative as possible, it is best to leave both burners adjusted the same and to burn continuously during the determination of a number of samples, and at the same time run a coke or coal standard. In this way if you do not make a standard what it should be, you can figure a factor from it and use on your other determinations. Make your standard coke or coal by having it analyzed by several different chemists and discard results that are extreme and take average of the balance.

METHOD FOR FIXED CARBON AND ASH

Burn two grams in a porcelain crucible over a burner, or best, in a muffle at a good red heat and weigh the ash. The sum of the percent. ash and per cent. volatile matter subtracted from 100 equals the per cent. of fixed carbon.

METHOD FOR ANALYSIS OF ASH

Burn off five grams and fuse ash with Na_2CO_3, dissolve in water, make acid with HCl and take to dryness. Take up in 20 cc. HCl and 25 cc. H_2O, boil, filter, ignite and weigh residue as SiO_2. Add a few drops HNO_3 to filtrate, make alkaline with NH_4OH, boil, filter and ignite precipitate over argand burner and weigh as Fe_2O_3 plus Al_2O_3. Fuse with a little Na_2CO_3, dissolve in HCl and titrate for iron as in ores. Calculate iron to Fe_2O_3, subtract from weight of Fe_2O_3 plus Al_2O_3, and the difference is your Al_2O_3. Now from the filtrate from the iron and aluminum hydrates, determine the CaO by adding five cc. $(NH_4)_2C_2O_4$ and evaporate to about 150 cc., filter and burn to CaO. Evaporate filtrate to about 200 cc., cool, add five cc. $(NH_4)_2HPO_4$, and then slowly with constant stirring 25 cc. NH_4OH, keep cold and stir frequently, let stand several hours, filter, wash with water containing one-fourth its volume of NH_4OH, burn in porcelain crucible and weigh as $Mg_2P_2O_7$. Calculate to MgO. Any Mn will be precipitated with the Mg.

METHOD FOR PHOSPHORUS

Ash five grams sample, fuse with Na_2CO_3, dissolve in water and HCl and take to dryness, add 10 cc. HCl, heat a few minutes and add 15 cc. HNO_3, heat until reaction is over, dilute with about 50 cc. H_2O, boil and filter into flask, add just enough NH_4OH to precipitate iron and aluminum hydrates, now add HNO_3, five cc. in excess of clearing solution, heat to 80° C and add 50 cc. molybdic solution, shake and let stand until precipitate settles, filter and wash well with cold water, place filter in flask, add 50 cc. H_2O free from CO_2, and 10 cc. standard NaOH solution, stopper flask and shake, wash off stopper into flask, add a few drops phenolphthalein and titrate with standard HNO_3 until pink color is just discharged. The difference between the cc. NaOH and HNO_3 multiplied by 0.4 is the phosphorus, if solutions are standardized for two grams iron or steel.

METHOD FOR SULFUR (ESCHKA'S METHOD)

Mix one gram of finely powdered sample with two and one-half grams of Eschka's mixture ($1Na_2CO_3$ to $2MgO$), burn slowly for some time and finally at a deep red heat, cool and digest with 100 cc. hot water and 10 cc. Br water, for one-half hour, filter and wash with hot water, add three cc. HCl, boil off Br, add 10 cc. $BaCl_2$ slowly, let stand at least one hour, filter, wash with hot water, ignite over argand burner and weigh as $BaSO_4$. Calculate to sulfur.

(PEROXID METHOD) ACCORDING TO PENNOCK AND MORTON

Place 0.7 gram of coke in a nickel crucible of about 30 cc. capacity and mix thoroughly with it, 11½ grams sodium peroxid by the use of a glass rod. Both should be finely pulverized and the peroxid should be dry. Cover the crucible with its cover, through which a small hole should have been drilled for the ignition of the contents. The crucible is now supported on a small strip of bent wire in a vessel of water so that about half of it is immersed. Ignite the contents by thrusting a red-hot wire through the hole in the cover into the mixture. After three or four minutes the crucible and cover are placed in a small beaker and about 50 cc. of water are added. The fusion will dissolve in a few minutes, when the crucible and lid are removed and rinsed off into the beaker. The solution is then made faintly acid with hydrochloric acid and filtered through a 9-cm. filter. The latter is washed thoroughly and the sulfur is precipitated in the boiling filtrate with barium chlorid in the usual way.

For coal use 0.7 gram of the sample and 16 grams of peroxid. Two things are essential in this method; first, the amounts of peroxid prescribed (11½ grams for coke and 16 grams for coal) must be taken and the mixture of coal or coke and peroxid must be intimate. If too little peroxid is used, the reaction will be of explosive violence; or if too much is employed, the combustion will be incomplete.

In the volumetric modification of the method, a tenth-normal sodium thiosulfate solution is made and also a solution of barium chromate in hydrochloric acid. The former is prepared by dissolving 24.8 grams of pure crystallized sodium thiosulfate in water and diluting to one liter. For the barium chromate solution, dissolve 23 grams of pure salt in a mixture of 80 cc. of concentrated hydrochloric acid and 920 cc. of water. Since the salt is difficult to obtain pure, Pennock and Morton recommend its preparation by adding dilute potassium chromate to the theoretical quantity of barium chlorid, also in solution, and then washing the precipitate thoroughly.

The ignition of the sample and peroxid and the solution of the resulting mass are affected as above, and the acidified solution is boiled for a few minutes. Ammonia is next carefully added until a few drops in excess, and the solution is again boiled a minute more. Then add 15 cc. of the barium chromate solution, and, after boiling a minute, dilute to 200 cc. and add a few drops of ammonia in excess of the quantity required to neutralize. Boil vigorously for a few minutes, filter and wash. Add one gram of potassium iodid crystals to the filtrate, cool to 30° C, add five cc. of hydrochloric acid and titrate with the thiosulfate solution, using starch indicator.

Cc. $N/10$ $Na_2S_2O_3 \times 0.153 =$ per cent. S.

Note: Standardize the $Na_2S_2O_3$ solution with a coke or coal in which the sulfur has been accurately determined gravimetrically.

LIMESTONE

SAMPLING

As the analysis of limestone from one location will sometimes vary a great deal, the sampling of same should receive careful attention. A sample taken from the top of a carload may not be at all representative of all the stone in the car, and, therefore, it should be sampled at time of unloading if possible. It is not at all necessary to take large pieces, but a large number of small pieces. If stone is coated with mud and clay, the sample should be washed and dried before preparing same for analysis, as it is always important to know the true analysis of stone. If stone shows much foreign matter a large sample should be washed and washings allowed to settle, when they should be dried and weighed. If there is a material amount the stone should be refused.

PREPARATION OF SAMPLE

If wet, dry the sample, then crush to one-half inch and mix well and crush a smaller portion to No. 8 size. This should be again well mixed and run through the grinder so most of it will pass a No. 20 sieve, mix well, and pass a smaller portion through the grinder so adjusted that the sample will pass a No. 80 sieve, mix well again and take a small sample for analysis.

METHOD FOR SILICA, ALUMINA, IRON, LIME AND MAGNESIA

One gram is mixed with one-half gram Na_2CO_3 and fused to a sintered mass. The crucible and contents are now transferred to a dish and treated with HCl (1.1), crucible is removed and solution taken to dryness. Now add 20 cc. HCl and 30 cc. H_2O, boil and filter, washing well with hot water. Ignite residue and weigh as SiO_2.

To filtrate, add a few drops HNO_3, heat to near boiling and make just alkaline with NH_4OH, boil about two minutes, filter and wash with boiled water, ignite on argand burner and weigh as Fe_2O_3 and Al_2O_3, fuse with Na_2CO_3, dissolve in HCl and titrate iron as in ores and from this calculate the Al_2O_3. When iron and alumina are present in small amounts they are not usually separated.

Heat the filtrate from the iron and alumina to the boiling point, add 50 cc. saturated solution of $(NH_4)_2C_2O_4$, diluted with 50 cc. water and also heated to boiling, add very slowly with constant stirring, and the precipitate will be very crystalline and wash easily, let stand until settled, filter and wash with hot water. It is very difficult to burn this to CaO, and, therefore, it is best to burn to $CaCO_3$ and partial CaO, cool, and add H_2SO_4 and heat until all H_2SO_4 is off, first over a low argand and finally on a burner at a red heat for one minute, cool and weigh as $CaSO_4$. Calculate to CaO or $CaCO_3$.

VOLUMETRIC MODIFICATION FOR CaO

Place filter containing the CaC_2O_4 in the beaker; now add while hot 50 cc. water containing 10 cc. H_2SO_4 and titrate at 60° C. with $KMnO_4$, which has been standardized by a standard limestone.

To filtrate from the CaC_2O_4, evaporated to 200 cc. and made acid with HCl, add 10 cc. $(NH_4)_2HPO_4$ after cooling. Now while stirring add slowly 25 cc. NH_4OH. Keep cool and let stand at least two hours, then filter and wash with water containing one-fourth its volume of NH_4OH, burn in porcelain crucible and weigh as $Mg_2P_2O_7$. Calculate to MgO or $MgCO_3$. Any manganese will be precipitated with the magnesia.

Note: If stone contains over 15 per cent. MgO the CaC_2O_4

should be dissolved in HCl and reprecipitated to free it from all magnesium. If limestone contains much organic matter it should be ignited before dissolving, when finishing the CaO by permanganate. Use boiled water and do not allow the alkaline solutions for the precipitation of lime to stand for any length of time, or the precipitate will contain $CaCO_3$ and results will be low by the $KMnO_4$ method. In dolomites make filtrate for MgO acid with HCl and evaporate to 300 cc. before precipitating the $Mg_2P_2O_7$.

METHOD FOR PHOSPHORUS

Dissolve five grams in 50 cc. HNO_3 (1.20), and boil 15 minutes, filter into flask, add 10 cc. NH_4OH. Heat to 80° C and add 50 cc. molybdic, shake and let stand until settled, filter and wash with cold water. Finish by titration with NaOH and HNO_3 as in phosphorus work on ores and cokes.

METHOD FOR SULFUR

Do same as in ores.

BLAST-FURNACE SLAG

SAMPLING AND PREPARATION OF SAMPLE

A sample is taken with a hand ladle and poured into a mould to cool. One sample should be taken from the middle of every flush during the 24 hours and a clean piece of the same size taken from each. These should be crushed quite finely, well mixed and a smaller portion then pulverized until it will pass a No. 80 sieve. Finally, mix well and take a small sample for analysis. If you wish to keep sample longer than one day it should be kept in a well stoppered bottle, as the sample will change its composition rapidly when finely pulverized.

METHOD OF ANALYSIS

Clean sample well with a magnet and then weigh 0.5 gram into a 4-inch porcelain dish, add 15 cc. H_2O, and then while stirring add 10 cc. HCl, cover and boil slowly over argand burner until HCl is mostly off, but not to dryness. Use an asbestos ring with a two and one-half-inch hole so as not to heat sides of dish, causing the silica to recombine and also adhere to dish so strongly that it is not easily removed. Now place in oven, heated to about

130° C, for one-half hour. Take up in 20 cc. HCl and 20 cc. H_2O, boil and filter at once, washing out dish and filter well with hot H_2O before removing the last of SiO_2 from dish. Ignite SiO_2 on blast-lamp for 15 minutes and weigh. Add 10 cc. NH_4OH to filtrate and then a few drops HNO_3, heat to boiling, add NH_4OH to just alkaline, boil about two minutes, let cool somewhat and also settle, filter and wash well with hot boiled water, ignite and weigh as Al_2O_3 plus any Fe_2O_3. If enough iron is present to color the Al_2O_3 to more than a deep yellow or light brownish color, it should be determined in a fresh sample, as in iron ores. *Caution.* —Boil off all H_2S before titrating for iron.

To the filtrate from the Al_2O_3 heated to boiling, add 30 cc. $(NH_4)_2C_2O_4$ (saturated solution diluted with equal volume of H_2O and heated to boiling) very slowly, with constant stirring, let settle, filter and wash with hot H_2O, place filter in beaker, add, while agitating, 10 cc. H_2SO_4 in 40 cc. H_2O and heat. Heat to 60° C and titrate with standard $KMnO_4$. The cc. $KMnO_4$ multiplied by the factor equal the CaO present.

To the filtrate, from the CaC_2O_4 evaporated to about 150 cc. and cooled and made acid by HCl, add 10 cc. $(NH_4)_2HPO_4$ solution and then while stirring, add 25 cc. NH_4OH, stir frequently, let stand two hours, filter and wash with H_2O containing one-fourth its volume of NH_4OH, burn in porcelain crucible until white and weigh as $Mg_2P_2O_7$. Calculate to MgO. If slag is high in Mg, do same as a dolomite. See method for limestone.

SULFUR

One-half gram in flask, add 75 cc. H_2O and five cc. starch solution and then a few cc. iodin solution, now add a few cc. HCl and then more iodin until blue color appears, now add more HCl, (in all about 50 cc.) and continue adding iodin until blue color remains permanent.

If iodin is standardized for three grams iron or steel, multiply cc. by 6 to get amount of sulfur in slag.

The results by this method will check very closely with those done gravimetrically.

Note: After removing the SiO_2, the Al_2O_3 may be done by the phosphate method as in ores.

If slag contains more than 15 per cent. MgO, the CaC_2O_4

must be dissolved in HCl and reprecipitated and both filtrates united for the MgO determination; otherwise, the CaC_2O_4 will retain a portion of the MgO. If slag contains manganese it will be precipitated as phosphate along with the Mg.

IRON

SAMPLING AND PREPARATION OF SAMPLES

In sampling iron from the runner while flowing from the furnace or cupola, a small amount should be taken by a hand ladle and poured into a mould 1×2×6 inches with a small projection at one end forming a tail piece about two inches long and one-half inch in diameter. The test is broken with a sledge and cooled. Drillings are now taken, being careful to reject any scale or other foreign matter from surface of test. In case the sample is too hard to drill, the small tail piece is pulverized by using a sledge and a case-hardened mortar and pestle.

In sampling from a pig, drill from bottom or from any point not above the center of the broken end. Drillings should always be clean and fine, in order to do good analytical work. In case of a hard iron, sieve the pulverized sample with a No. 60 sieve, and use only the portion which passes through.

SULFUR

Do exactly as in steel, except use hot HCl, instead of cold, in dissolving.

SILICON

0.94 gram is weighed into a five-inch porcelain dish and covered by a watch glass, 25 cc. silicon mixture is added and heated until all iron is in solution and fumes of SO_3 come off, cool and add 10 cc. HCl (1.1) and 30 cc. H_2O, boil and filter at once, wash once with H_2O, then once with hot HCl (1.1) and finally well with H_2O, ignite until white and weigh as SiO_2. Calculate to Si.

PHOSPHORUS

Two grams in erlenmeyer flask, add 50 cc. HNO_3 (1.20) and eight cc. NH_4OH, boil slowly until all iron is dissolved, adding a little water from time to time, so as not to let solution evaporate below 50 cc., filter into another flask, add 10 cc. $KMnO_4$ and boil five minutes, add a few drops of sugar solution and boil until clear, cool to 80° C, and add 50 cc. molybdic, shake and let stand until settled, filter on suction and wash well with two per cent. HNO_3, and then well with H_2O. Transfer filter to flask, add 50 cc. H_2O free from CO_2 and enough standard NaOH solution to more than dissolve stopper and shake, wash off stopper into flask, add a few drops phenolphthalein and titrate with standard HNO_3 until pink color just disappears. Note the number of cc. NaOH used in excess of the HNO_3. One cc.=0.01 per cent. phosphorus.

Note: When sample contains much combined carbon, add more $KMnO_4$, and at two different times.

MANGANESE

One gram is dissolved in 10 cc. H_2O and 20 cc. HNO_3 (1.20), diluted to 100 cc. (exactly) and well mixed, draw off 20 cc. with pipette into a small flask, add 20 cc. HNO_3+AgNO_3, heat to boiling, remove from heat and add 15 cc. $(NH_4)_2S_2O_8$ solution, shake, let stand for a few minutes, cool, add seven cc. NaCl solution, and titrate with standard arsenite.

Note: Arsenite should be standardized with a standard iron or steel in the same way as sample is run. See notes under manganese in steel, all of which will apply to iron work as well.

GRAPHITIC CARBON

One-half to one gram is dissolved in HNO_3 (1.13), being careful to heat until all nitrated carbon is in solution, filter on boat containing ignited asbestos, wash well with HCl (1.1) and water, then with NaOH solution, then with H_2O, then with alcohol, then with ether, then with cold H_2O, and finally with hot H_2O until ether is removed. Finish by combustion in platinum tube with oxygen, precipitating the CO_2 as $BaCO_3$ with a solution of $Ba(OH)_2$.

Note: See method for determining carbon in iron and steel by combustion, page 33.

The graphite, after the above treatment, may be dried at 100° C, weighed and burned. The loss is the graphite plus any combined water and sulfur.

COMBINED CARBON

Do total carbon by the combustion method. The difference between total carbon and graphitic carbon is the combined carbon.

TOTAL CARBON

See method for determination of carbon in iron and steel by combustion, page 33.

Note: In doing graphite in white irons and in ferro-silicon, use HCl (1.1) instead of HNO_3 (1.13), and finish by combustion. When gelatinous SiO_2 separates, a few drops of HF will dissolve and aid in filtering.

STEEL

SAMPLING AND PREPARATION OF SAMPLES

The most reliable results are always obtained from a small sample taken by a hand ladle during the pouring; this should be poured into a small mould of such dimensions as to form a test-piece about 1×2×6 inches. This is small enough so as not to cause much segregation of the metalloids. The test should be allowed to cool in the air. If chilled by water, the carbon results by the color method will be too low. The test mould is made in two pieces and is poured at the end. One side of the test can be used for stamping the number on, and drillings can be taken from the other side.

Owing to segregation, it is next to impossible to take drillings from a large ingot or other steel castings that are representative of the steel. The same is true to a certain extent in blooms, billets, bars and plates. Blooms and billets should be drilled from surface to center and sample well mixed. Bars and plates should be drilled half way through and midway between the longitudinal center and the outer edge. Drillings may also be taken from various places and separate analysis made of each, or if drillings are very fine, the whole can be mixed and one analysis made. Sheets and all material too thin to take samples by drilling should be cleaned by HCl, washed and dried quickly, then sheared up into small pieces by a heavy pair of shears. Tin can be removed from sheets with HNO_3.

METHODS OF ANALYSIS—CARBON (COLOR METHOD)

Weigh 0.2 gram into a ⅝×5-inch test-tube, add five cc. HNO_3 (1.20) and place in water-bath, heat at boiling point until all nitrated carbon is dissolved, which will take from five to 20 minutes, for C from 0.07 to 0.12 per cent. Cool and compare in camera with white glass plate, behind which is an arc light or a mantle gas light. Use comparison tubes graduated in cc. only and with first six cc. at bottom of tubes left blank. Pour and wash standard into tube, dilute with H_2O so that each cc. will be equal to 0.01 carbon, mix well by closing end of tube and inverting several times. Treat sample in same way, comparing until colors match, when the reading in cc. will be carbon. One cc.=0.01 carbon.

Note: The smallest amount of sample that should be used in any case is 0.2 gram, and in comparing colors, this should always be diluted to as many cc. as there are hundredths per cent. carbon.

In steel containing not over 0.12 carbon, the segregation is so small that it is not difficult to make a standard that is uniform when using as little as 0.2 gram sample, but in doing all higher carbons use not less than 0.5 gram. In this case, however, dilute to two or three times the volume you do when using 0.2 gram, and then take one-half or one-third of your final volume as the per cent. carbon. This procedure will make any error in comparing colors very small, and is a much better way than the practice in many places of diluting to only one-half and then multiplying your reading by two, in which case you are doubling any error.

In all cases where carbon is above 0.10, I would recommend using a hot-plate instead of water-bath and a small erlenmeyer flask instead of the test-tubes, and use from 10 to 25 cc. of HNO_3 (1.20), according to whether you are doing low or high carbons. Have plate uniformly heated and to such a temperature that it will just boil the solutions. Let boil for about one minute after steel is in solution.

This method of procedure is more rapid than the water-bath, and properly handled, it is just as good.

In all cases, use same amount of acid on standard as on samples, have all dissolve with same rapidity, subject them to like degrees of heat for a like period of time, and compare all without delay, and results should be comparative and quite accurate.

In all cases use a standard which contains very nearly the same amount of carbon as the sample being analyzed.

The steel from which standards are made, must have been made by the same process, and also must have undergone the same subsequent treatment as nearly as possible, as the samples to be analyzed.

In all cases where you do not have standards to conform to the above, and also where you do not know the method of making, and the treatment of the steel to be analyzed, the determination should be made by the combustion method.

For combustion method, see page 33.

MANGANESE

Weigh 0.2 gram into a small flask, add 15 to 25 cc. HNO_3 plus $AgNO_3$ solution, heat until dissolved and all N_2O_4 disappears, remove from heat and add five to 15 cc. $(NH_4)_2S_2O_8$, shake up and let stand several minutes, cool, dilute to about 50 cc. and titrate with standard arsenite solution.

Note: Fifteen cc. HNO_3 plus $AgNO_3$ and five cc. $(NH_4)_2S_2O_8$ are enough for low carbon steels and where manganese is not over 0.75 per cent. For high carbon steel and those containing over 0.75 manganese, more of each solution must be used.

There is no appreciable reoxidation of the manganese during titration, providing solution is cold and not too large an excess of persulfate.

To prevent any reoxidation, add just before titrating, seven cc. of 0.2 per cent. c. p. NaCl solution.

SULFUR

Three grams are placed in a 300 cc. erlenmeyer flask fitted by a stopper, carrying a straight funnel-tube and a bridge of glass tubing. The bridge is so made as to reach the bottom of a 1×9-inch test-tube which is half filled with caustic solution and placed in a rack. Add about 100 cc. HCl (1.1) and heat slowly until all is in solution, then bring to a boil until all H_2S is expelled from flask. Remove flask from light, empty contents of test-tube into a glass tumbler, fill test-tube with water and pour into tumbler, add about 30 cc. HCl (1.1) and titrate, while stirring, with standard iodin until the blue color appears. One cc. iodin=0.01 sulfur.

PHOSPHORUS

Place two grams steel in an erlenmeyer flask, add 50 cc. HNO_3 (1.20) and eight cc. NH_4OH, heat, and when in solution, add 10 cc. $KMnO_4$ solution, boil about five minutes, add a few drops of a sugar solution and boil until clear, dilute with H_2O to about 80 cc., bring to 80° C, add 50 cc. molybdic, shake and let stand until settled, filter, wash well with two per cent. HNO_3, and then well with cold water. Now transfer filter to flask, add about 50 cc. of H_2O free from CO_2, and enough standard NaOH to more than dissolve, stopper flask and shake, wash off stopper into flask, add a few drops of phenolphthalein and titrate with standard HNO_3 until pink color just disappears. The difference in number of cc. HNO_3 and NaOH used is the phosphorus. One cc.=0.01 phosphorus.

Note: For steel which is high in carbon, use more $KMnO_4$ and add it at two different times.

SILICON

Five grams steel in porcelain dish, add 20 cc. HCl and 15 cc. H_2O, heat slowly until in solution, then boil rapidly to dryness, remove cover glass and heat slowly until $FeCl_2$ begins to turn brown, cool, add 20 cc. HCl and afterwards about 80 cc. of water, boil, filter, wash well with HCl (1.1) and water, ignite and weigh as SiO_2. Calculate to Si.

Note: The SiO_2 will retain a little iron, and if you wish to be accurate, finish by volatilizing with HF and a drop of H_2SO_4.

GRAVIMETRIC METHOD FOR MANGANESE IN FERRO-MANGANESE

Three-tenths to 0.5 gram are dissolved in 35 cc. HNO_3 (1.20), evaporated to dryness and baked until all N_2O_4 fumes are off. Take up in 25 cc. HCl, and boil off all Cl, dilute to about 150 cc. with H_2O, add NH_4OH until a slight precipitate forms, now add $NH_4C_2H_3O_2$ until all iron is precipitated, boil and filter, washing twice with hot water, dissolve precipitate in 15 cc. HCl and precipitate again as before. Repeat the solution and precipitation, making three precipitations in all. This should remove all manganese if the process has been properly carried on. Add 50 cc. $(NH_4)_2HPO_4$ solution to the filtrates, clear with HCl and heat to boiling; now add NH_4OH slowly with constant stirring until all Mn is precipitated in a granular form, make strongly alkaline with NH_4OH, cool, filter, and wash with H_2O, ignite and weigh as $Mn_2P_2O_7$. Calculate to Mn.

GRAVIMETRIC METHOD FOR MANGANESE IN IRON ORES, IRON, STEEL, AND SPIEGEL

The HCl solution of one gram sample, from which the SiO_2 has been removed, is boiled for several minutes with a few cc. HNO_3 and a basic acetate separation made as follows: Dilute with cold water to about 200 cc. and add NH_4OH until a slight permanent precipitate is formed. The solution is now diluted to about 400 cc., and 25 cc. of a 20 per cent. solution of $NH_4C_2H_3O_2$ added and heated to boiling, and boiled about one minute. Now filter through a large filter and wash once with hot water. In case the manganese is above one per cent., redissolve in HCl, and make another basic acetate precipitation, keeping solutions about one-half the size as before. Heat filtrates to boiling, add 20 cc. NH_4OH and one gram $(NH_4)_2S_2O_8$, and boil about 10 minutes, filter and wash well with hot H_2O, burn off paper and then ignite strongly and weigh as Mn_3O_4. Calculate to Mn.

Note: The Mn_3O_4 is liable to contain some impurity. Any correction for SiO_2 present can be made by dissolving in HCl, filtering and igniting and weighing the residue.

A more accurate method, however, is to dissolve in HCl and dilute to 50 cc. and make a basic acetate separation, which will remove any iron, alumina and silica. Then reprecipitate as before with $(NH_4)_2S_2O_8$, or finish as $Mn_2P_2O_7$, as follows. Add five or 10 cc. $(NH_4)_2HPO_4$ solution, make strongly alkaline with NH_4OH, and boil until precipitate is crystalline. Now filter and wash with hot H_2O, ignite and weigh. Calculate the $Mn_2P_2O_7$ to Mn.

GRAVIMETRIC METHOD FOR PHOSPHORUS

For iron or steel dissolve in HNO_3 (1.20), take to dryness and bake well over an argand burner, take up in 25 cc. HCl and evaporate to about five cc., add 10 cc. HNO_3 while still hot, dilute with H_2O and filter into flask, add 10 cc. NH_4OH, shake up and add 10 cc. HNO_3 to clear, heat to 80° C and add 50 cc. molybdic, when settled, filter and wash with two per cent. HNO_3 and then with H_2O. Finish by drying for one hour at 110° C and weigh. Determine factor by doing a standard iron or steel in exactly the same manner, or finish by the magnesia method as follows: Dissolve the yellow precipitate into a flask by adding a few drops of

NH_4OH and then washing out with a little H_2O, make just acid with HCl, and then add just enough NH_4OH to clear, pour again through filter to remove any SiO_2, wash well, cool and add 10 cc. magnesia mixture and then 10 cc. NH_4OH, let stand two hours and filter, washing with water containing one-fourth its volume of NH_4OH, burn in a porcelain crucible and weigh as $Mg_2P_2O_7$. Calculate to P.

Ores, cokes, limestone, etc., may be done for phosphorus by the same methods by removing all SiO_2 from solution before precipitating phosphorus with molybdic. Use precaution for As and Ti as in other methods. In doing phosphorus by weighing the yellow precipitate, it is very important that the temperature of drying should always be the same and that the factor should be determined from running a standard.

In finishing by the magnesia method, large samples should be worked with, thereby reducing any error to a minimum. For example, 0.001 gram $Mg_2P_2O_7$ is equal to 0.0055 per cent. P when working on five grams sample.

GRAVIMETRIC METHOD FOR SULFUR IN IRON AND STEEL

Dissolve from three to five grams in HNO_3 (1.42), add 0.5 gram sulfur free Na_2CO_3 and evaporate to dryness in porcelain dish, and bake until all nitrate is converted into the oxid. Cool and take up in from 20 to 40 cc. HCl, dilute with equal amount of H_2O and filter. Now add NH_4OH in slight excess, bring to boiling, add 10 cc. $BaCl_2$ solution and boil a few minutes. Now add HCl until all iron is in solution and a few cc. excess of HCl. Cool thoroughly and add dilute NH_4OH until red color begins to form, then HCl, two cc. in excess of amount necessary to destroy red color. Bring to boiling again and evaporate to about 100 cc. Let stand 12 hours and filter, washing with cold H_2O, then with a wash made of equal parts HCl and $BaCl_2$ solution with water added until solution is clear. Then wash again with cold H_2O. and finally with hot H_2O. Burn on argand or bunsen and weigh as $BaSO_4$.

DETERMINATION OF CARBON IN IRON AND STEEL BY COMBUSTION

From one to five grams of the clean and finely divided sample is placed in a beaker and the $(KCl)_2CuCl_2$ solution (50 cc. for each gram sample) added. Cover by a watch glass with hole in center and stir constantly by a mechanical stirrer until all is in solution. In the absence of a good mechanical stirrer operated by a small electric or water motor, a blast of air can be used, by cleaning the air well by first passing it through a tube packed with absorbent cotton and then allowing it to bubble through solution. Solution is more rapid if heated, but should not be heated to above 50° C. When all precipitated copper is in solution, filter on a platinum boat containing a pad of ignited asbestos, washing beaker and boat with HCl (1.1), and then well with hot H_2O, use suction, and continue after washing, until most of the water is removed. Transfer to platinum tube of combustion furnace, placing it over the second and third burners, connect all parts of the combustion train, and start the oxygen gas or air, as the case may be, to bubbling through at the rate of two to four bubbles per second having 100 cc. of hot $Ba(OH)_2$ solution in the 10 bulb tube. Now light the burners under end of tube containing the CuO, when this is at red heat gradually turn on burners one by one until all are lighted, beginning at end next to CuO. If using oxygen, pass it for 10 minutes and then continue with air for 10 minutes longer. Now stop the flow of air, turn out lights and disconnect Meyer bulb containing the $BaCO_3$, filter rapidly on a double filter, using suction and washing with boiled water which has partially cooled. Burn on argand and finally on bunsen and weigh. Calculate $BaCO_3$ to C.

Note: The combustion train consists of the following parts beginning at the oxygen cylinder end. A bottle of NaOH solution (100 grams NaOH to 200 cc. H_2O) through which the air or oxygen is passed, a bottle of stick NaOH, the platinum tube containing CuO at the end nearest the Meyer bulb. The CuO is made by rolling loosely a strip of Cu gauze and heating in a muffle until it is all burned to CuO. This can be broken up or can be placed in tube as it is, and is held in place by a coil of platinum gauze placed on each side of it. Next is a small glass bulb to catch any water, etc., that has been retained in the boat. Last of all is the 10-bulb tube for the absorption of the CO_2.

For burning the carbonaceous residue from steels, air alone is just as good as O, but for all combustions containing graphitic carbon, oxygen should be used, also for direct combustion of alloys and iron and steel without solution of the metal. For aspiration of the air, aspiration bottles can be used or a uniform water or steam suction can be used.

By a little experience the $Ba(OH)_2$ can be so handled as not to allow it to come in contact with the larger bulb of the Meyer apparatus, thereby doing away with the precipitation of $BaCO_3$ on the inner surface of this bulb which is sometimes difficult to remove.

Ferro-Manganese and Spiegel: Mix with ZnO and do by direct combustion with oxygen.

Ferro-Silicon and Silico: Spiegel by direct combustion of the finely powdered substance without any admixture.

The CuO should be well ignited now and then by passing O for sometime at a high temperature. This will decompose any CuS which may have been formed and which is very injurious to the platinum. When doing steel containing Cr, wash the C residue with H_2O only, as HCl sets free hydrocarbons.

DETERMINATION OF PHOSPHORUS IN ORES, IRON AND STEEL CONTAINING ARSENIC

The following method practically as given by J. M. Camp: The method given is for ores and the chemist can readily apply it to iron or steel.

Five grams are dissolved in 50 cc. HCl. When solution of iron is complete, dilute with H_2O and filter. The filtrate will contain all the non-volatilized As in the ore. Fuse residue, dissolve in H_2O in separate beaker, make acid with HCl. Both are now taken to dryness. Add two grams oxalic acid ($H_2C_2O_4$) to beaker containing the Fe_2Cl_6 and then 50 cc. HCl and boil to dryness, but do not bake, add 30 cc. HCl and evaporate again until Fe_2Cl_6 begins to separate, add 10 cc. HNO_3, heat until reaction is over, add 10 cc. HCl to the beaker containing the fusion, heat until most of the HCl is off, add 10 cc. HNO_3, and heat again, dilute with enough H_2O to dissolve the salts, boil and filter along with the other portion, finish as other phosphorus in ores.

FERRO-MANGANESE

SAMPLING AND PREPARATION OF SAMPLE

When sampling from a carload, use a heavy sledge and break the larger pieces until you can get a clean piece without containing any surface oxidation. Take a great number of small pieces of equal size from every part of the car load, crush fine enough to pass a No. 20 sieve, mix well and take about 500 grams and pulverize in the same mortar until it will all pass a No. 60 sieve, take a portion of this for analysis.

METHODS OF ANALYSIS, SILICON

Do same as in iron.

PHOSPHORUS

Five-tenths gram, dissolve in 35 cc. HNO_3 (1.20) in a porcelain dish, evaporate to dryness and bake over argand burner, cool and add 10 cc. HCl, boil until most of the HCl is off, add 10 cc. HNO_3, heat, dilute with 50 cc. H_2O and filter, ignite residue and fuse with little Na_2CO_3, add fusion to the solution, heat to 80° C and add 50 cc. molybdic.

Finish like all other phosphorus.

CARBON

Mix 0.5 gram of the finely powdered sample with about two grams ZnO and place in a porcelain boat. Do by combustion in oxygen in a platinum tube as in iron and steel. Do a blank on the ZnO to see whether it contains any C, if so ignite it previous to adding the ferro-manganese.

MANGANESE

One-half gram is dissolved in 35 cc. HNO_3 (1.20), cool to temperature of the laboratory and add to a flask graduated at 500 cc. washing in with water at same temperature, add ZnO to slight excess and then fill to the mark with the H_2O, pour into a larger flask and shake well, let settle, pour off 250 cc., and heat to boiling and titrate with standard $KMnO_4$ until the last drop causes a pink color.

Standardize $KMnO_4$ with a standard ferro-manganese.

FERRO-SILICON FOR Si

LOW GRADE, 10 PER CENT. OR LESS

Dissolve 0.5 gram in HCl plus HNO_3, when in solution add 10 cc. H_2SO_4 and evaporate to fumes of SO_3, cool, dilute with H_2O

and a few cc. HCl, filter and wash with H_2O and hot HCl (1.1), burn slowly for sometime and finally ignite on blast-lamp until white and weigh as SiO_2. Calculate to Si.

HIGH GRADE

Mix 0.5 gram with six to eight grams sodium peroxid (Si free) and fuse in nickel crucible, heat very slowly until fusion begins, then heat to dull redness, cool and dissolve in about 50 cc. H_2O, wash out crucible and make acid by adding 10 cc. dilute H_2SO_4 and finally make strongly acid with HCl.

Evaporate until fumes of SO_3 come off freely, cool, add about 50 cc. H_2O and a little HCl, boil and filter at once, washing well with HCl (1.1) and hot water. Evaporate filtrate to fumes and take up as before and filter to remove all the SiO_2, add this to the first SiO_2 and burn off slowly, finally ignite strongly and weigh, treat with H_2SO_4 and HF, ignite and weigh again.

Calculate SiO_2 to Si.

Note: Six grams Na_2O_2 are enough for samples below 50 per cent. Si, but for those much higher, more should be used or the combustion may be of explosive violence.

Some samples contain a small amount which is infusible by the one treatment with Na_2O_2. Filter it off, ignite and fuse in platinum crucible with Na_2CO_3 and add to the acid solution.

SLAGS AND CINDERS

CUPOLA, LADLE, VESSEL AND OPEN HEARTH SLAGS
MILL AND TAP CINDER, SCALE
SAMPLING

If sampling from an open hearth furnace, take a sample with a hand ladle; if from a ladle or vessel, sample slag thoroughly after it is dumped from the container after the metal has been poured.

Mill and tap cinder are usually sampled from the car or stock pile; take a great number of small pieces from every part of carload or stock pile.

Crush as finely as possible in a small crusher or if they contain shot iron, crush by hand in a mortar. If containing shot iron now remove same with a magnet, mix sample thoroughly and pulverize a small portion so it will pass a No. 80 sieve. Take a small portion and if moist, dry it for analysis. This sample may be now cleaned of all metallic iron by use of magnet and by weighing all the metallic iron removed and also the slag, the percentage of each can be calculated.

METHOD FOR ANALYSIS

The above slags, cinders, and scale are very similar to iron ores in composition. For methods of analysis, use those given under ores with the following exceptions:

If sample contains any metallic iron, in making phosphorus determinations, moisten with water and then with HNO_3, and heat for a few minutes before adding HCl. In this way you will not lose any phosphorus. It may also be necessary to take HCl solution to dryness before filtering and fusing residue, otherwise the gelatinous SiO_2 will prevent filtering.

Some samples and especially basic open hearth slags will contain CrTi and V, and it is well to observe precautions for same. As the manganese is high in most of these, the gravimetric method will be found best for this determination.

METHODS FOR PHOSPHORUS IN BASIC OPEN HEARTH SLAGS

One-tenth to one gram, according to whether phosphorus is high or low, is stirred up well with 10 cc. water and then, while stirring, add 20 cc. HCl and a few drops HNO_3, boil to light dryness, add 20 cc. HCl and 20 cc. H_2O, boil and filter add a little Fe_2Cl_6 (enough to make a deep red $Fe_2(OH)_6$ precipitate when adding NH_4OH), heat to near boiling and add NH_4OH until a slight precipitate, now add a few cc. of a 20 per cent. solution of $NH_4C_2H_3O_2$, boil and filter. This precipitate, if containing enough iron, will carry all phosphate as ferric phosphate. Dissolve precipitate into flask with 30 cc. hot HNO_3 (1.20), add 10 cc. NH_4OH, heat to 80° C and add 50 cc. molybdic, finish as any other phosphorus work.

METHOD FOR ANALYSIS OF FLUORSPAR

SILICA

Decompose 0.5 gram in platinum crucible with H_2SO_4 and evaporate off all excess acid and heat until no more fumes come off, add five grams Na_2CO_3 and fuse for one-half hour, dissolve in HCl and evaporate to dryness, take up in 35 cc. HCl plus 35 H_2O, boil and filter, washing well with HCl (1.1) and hot H_2O. Ignite and weigh the SiO_2. Decompose the SiO_2 with HF and a few drops H_2SO_4, ignite and weigh as it may contain much impurity.

ALUMINA, IRON OXID, LIME AND MAGNESIA

These are determined in the filtrate from the SiO_2 exactly as in a limestone. If there is any CO_2 present it is determined by decomposing with $H_2C_4H_4O_6$ and precipitating as $BaCO_3$, filtering, washing with hot H_2O, igniting and weighing. Calculate $BaCO_3$ to CO_2. From the total CaO, subtract the amount necessary to combine with the CO_2 and calculate the balance to CaF_2.

Note: The above method is practically as worked out by Mr. E. J. Ericsson and published in "Iron Age," of August 27, 1903. The author's experience with the method is very satisfactory, but the following points are worthy of notice: The sample must be completely decomposed by H_2SO_4, for if any F is present it will precipitate as CaF_2 along with the Fe and Al.

When igniting and weighing the SiO_2 it is necessary to use HF or with some samples the results will be far from correct.

Old method for determination of SiO_2. Treat one gram with cold dilute HCl (one of HCl to 10 of H_2O), filter and wash with H_2O, ignite slowly at a low heat until paper is just burned off, cool and weigh, treat with a little HF, heat to drive off all excess HF, cool and weigh again. The difference is the SiO_2.

FIRE-STONE, FIRE-SAND, GANISTER, MICA SCHIST, ETC.

The sample must be finely pulverized, by pounding in a manganese steel mortar, being very careful not to contaminate the sample by wearing off of mortar and pestle. After a degree of fineness is reached so all will pass a No. 60 sieve, a small portion can be finished in agate.

METHODS OF ANALYSIS—SILICA

Five-tenths gram of the well dried sample is fused with 5 grams Na_2CO_3, dissolved in H_2O, made acid with HCl and taken to dryness. Fifty cc. H_2O and 25 cc. HCl are now added and boiled, filtered and washed well with hot H_2O. If accuracy is desired, filtrate must be again evaporated to dryness and taken up as before and filtered to remove all SiO_2. The SiO_2 is now ignited and weighed.

ALUMINA AND IRON OXID

To filtrate from SiO_2, add a few drops HNO_3 and heat to boiling, add NH_4OH to just alkalinity, boil, filter and wash well with hot boiled H_2O, burn on argand and weigh as Fe_2O_3 plus Al_2O_3, fuse with Na_2CO_3, and dissolve in HCl, precipitate with NH_4OH, filter, dissolve in HCl and titrate for Fe as in ores, calculate Fe to Fe_2O_3. Subtract the Fe_2O_3 from the Fe_2O_3 plus Al_2O_3 to find the Al_2O_3.

LIME

To filtrate from the above, add five cc. $(NH_4)_2C_2O_4$ solution and evaporate to 150 cc., add 10 cc. NH_4OH and boil, filter and wash with hot H_2O, ignite and weigh as CaO.

MAGNESIA

To the cold filtrate from the CaO, add five cc. $(NH_4)_2HPO_4$ solution, make acid with HCl and then add slowly, while stirring, 25 cc. NH_4OH, let stand several hours, filter and wash with H_2O plus one-fourth volume of NH_4OH, ignite in porcelain crucible and weigh as $Mg_2P_2O_7$. Calculate to MgO.

Note: If there is any Mn present it is removed by adding two grams of $(NH_4)_2S_2O_8$ to filtrate from Fe_2O_3 plus Al_2O_3 and digesting for several hours, and filtering, igniting and weighing as Mn_3O_4. In this case, however, the iron and alumina should be precipitated by basic acetate method.

FIRE-BRICK, CLAYS AND KAOLIN

Clays and kaolin are very easily pulverized, but on account of the hardness of fire-brick, extreme care must be used in pulverizing so as not to contaminate sample by the wearing off of the utensils used.

METHODS OF ANALYSIS—SILICA

Five-tenths gram of the dried sample is fused with five grams Na_2CO_3, dissolved in H_2O and made acid with HCl. Take to dryness, add 50 cc. H_2O and 25 cc. HCl, boil and filter, washing well with hot H_2O. If accuracy is desired, take filtrate to dryness and take up in HCl and H_2O and filter as before to remove all SiO_2, ignite SiO_2 and weigh.

ALUMINA AND IRON OXID

To filtrate, add a few drops HNO_3, boil and precipitate with NH_4OH, filter and burn on argand and weigh as Fe_2O_3 plus Al_2O_3, fuse with Na_2CO_3, dissolve in HCl, precipitate with NH_4OH and filter, dissolve in HCl and titrate Fe as in ores, calculate to Fe_2O_3, subtract from Fe_2O_3 plus Al_2O_3 to find Al_2O_3. Remove any Mn as in fire-stones, etc.

LIME

To filtrate, add five cc. $(NH_4)_2C_2O_4$ and evaporate to 150 cc., add 10 cc. NH_4OH and boil, filter, ignite and weigh as CaO.

MAGNESIA

To cold filtrate from CaO, add five cc. $(NH_4)_2HPO_4$, make acid with HCl, and slowly, while stirring, add 25 cc. NH_4OH, let stand several hours and filter, washing with H_2O plus one-fourth volume NH_4OH, burn in porcelain crucible and weigh as $Mg_2P_2O_7$. Calculate to MgO.

COMBINED WATER

One gram is ignited at a red heat until there is no further loss in weight. The loss is combined water.

SULFUR TRIOXID

The SO_3 is determined exactly as in iron ores.

TITANIUM

This may be determined the same as given for Ti in ores, iron and steel.

POTASSIUM AND SODIUM

As these are rather difficult determinations and require experience in the hand of any operator, I will give the two standard methods, either of which give equally good results. Platinum dishes should be used and a blank run with all reagents to determine their purity.

First Method: One gram is digested with 10 cc. H_2SO_4 and 20 cc. HF until all is dissolved, then heated until excess of acid is driven off. Dissolve in 20 cc. HCl and 25 cc. H_2O, add few drops HNO_3 and boil, add NH_4OH until alkaline, boil, filter off Fe_2O_3 plus Al_2O_3, to filtrate, remove lime with $(NH_4)_2C_2O_4$, letting stand several hours until all is precipitated, filter and remove MgO in the usual way with $(NH_4)_2HPO_4$. The filtrate is evaporated to dryness and ignited at just a visible red heat to expel all NH_4 salts. A few drops HCl are added and evaporated again to dryness. Dissolve in a little water, add excess of platinic chlorid and evaporate nearly to dryness, add 20 cc. 80 per cent. alcohol and let stand until all Na salts are dissolved, filter on a weighed paper and wash with 80 per cent. alcohol, dry and weigh as K_2PtCl_6. Calculate to K_2O. The filtrate is evaporated to dryness and the platinic salts decomposed by heating to redness with $H_2C_2O_4$, boiled with H_2O, filtered, a few drops H_2SO_4 added and evaporated to dryness and ignited carefully to constant weight and weighed as Na_2SO_4. Calculate to Na_2O.

Second Method: Grind one gram with one gram NH_4Cl and mix with eight grams $CaCO_3$, heat in platinum crucible to redness for an hour. Digest in hot H_2O until all is disintegrated, filter and wash well with hot H_2O. To filtrate, add NH_4OH and $(NH_4)_2CO_3$ in excess, heat, filter and wash with H_2O and a little NH_4OH, evaporate filtrate to dryness and ignite at just a visible red until all NH_4Cl is expelled and no more fumes form. Add a little H_2O and a few drops $(NH_4)_2CO_3$ and filter off any precipitate, add a few drops HCl, and evaporate to dryness and ignite carefully as before and weigh as KCl plus NaCl. Dissolve in H_2O, add excess of platinic chlorid, evaporate nearly to dryness, add 20 cc. alcohol (80 per cent.), let stand until Na salts are dissolved, filter on weighed paper, wash with alcohol (80 per cent.), dry and weigh as K_2PtCl_6. Calculate to K_2O. Also calculate to KCl, subtract from the KCl plus NaCl and then calculate the NaCl to Na_2O.

Note: For method for determination of free and combined silica, consult "Engineering Chemistry," Stillman.

PORTLAND CEMENT

METHOD OF ANALYSIS—SILICA

Stir 0.5 gram of the dried sample with 15 cc. H_2O and then add 15 cc. HCl, take about to dryness and heat in an oven at about

130° C for one-half hour as in slag analysis. Take up in 20 cc. HCl and 25 cc. H_2O, boil and filter, washing well with hot H_2O. Ignite and weigh the SiO_2.

Note: If cement is not completely soluble in HCl, the residue should be fused and added to main solution before evaporating to dryness.

ALUMINA AND IRON OXID

To the filtrate from the SiO_2 heated to boiling, add a few drops of HNO_3 and then make just alkaline with NH_4OH, boil about one minute and filter, washing well with hot boiled H_2O, ignite on argand and weigh as Al_2O_3 plus Fe_2O_3. Dissolve one gram of the cement in HCl and titrate for Fe as in ores, calculate to Fe_2O_3, divide by two and subtract from the weight of Al_2O_3 plus Fe_2O_3 to find Al_2O_3.

LIME

To filtrate from the above heated to boiling, add slowly 35 cc. $(NH_4)_2C_2O_4$ which has been diluted somewhat with H_2O, and also heated to boiling, let stand until settled, filter and wash with hot H_2O, wash CaC_2O_4 into flask, add 10 cc. H_2SO_4 diluted with 50 cc. H_2O, heat to 60° C and titrate with $KMnO_4$.

MAGNESIA

Evaporate filtrate from the CaO to about 150 cc., cool and add five cc. $(NH_4)_2HPO_4$, make acid with HCl, and then slowly, with stirring, add 25 cc. NH_4OH, let stand several hours, filter and wash with H_2O containing one-fourth volume of NH_4OH, ignite in porcelain crucible and weigh as $Mg_2P_2O_7$. Calculate to MgO, which should not exceed five per cent.

SULFUR TRIOXID

Dissolve one gram in HCl and take to dryness to remove the SiO_2. Take up in five cc. HCl and 50 cc. H_2O, filter and wash with hot H_2O. Heat filtrate to boiling and add 10 cc. $BaCl_2$, let stand until all $BaSO_4$ is precipitated, filter, washing well with hot H_2O, and burn on argand, weigh as $BaSO_4$. Calculate to SO_3, which should not exceed 2.5 per cent.

SODIUM AND POTASSIUM

If these are desired determine same as in clays.

SPECIFIC GRAVITY

Take with Le Chatelier's apparatus, being careful to have a constant degree of temperature.

Note: For the mechanical testing of cement consult "Engineering Chemistry," Stillman.

CHROMIUM AND TUNGSTEN STEELS

The following method is used and is practically the same as that by A. G. McKenna.

If steel is too hard to drill, crush in a mortar and sieve on a No. 60 sieve.

SULFUR

Do same as in regular analysis of steel by the evolution method.

SILICON AND TUNGSTEN

Five grams in a porcelain dish, add 30 cc. HCl and 20 cc. H_2O, boil until dissolved, add 10 cc. HNO_3 and evaporate to dryness, add 20 cc. HCl and heat until all iron is in solution, and evaporate again to dryness to render all WoO_3 insoluble. Take up again in HCl, heating slowly until dissolved, dilute to 100 cc. with H_2O, boil and filter off the SiO_2 plus WoO_3, wash well with HCl (1.1) and H_2O, ignite and weigh as SiO_2 plus WoO_3, add a few drops H_2SO_4 and a little HF, evaporate off acid, ignite and weigh again. The loss is SiO_2, which calculate to Si. The residue is WoO_3 and a little Fe_2O_3, fuse with a little Na_2CO_3, dissolve in H_2O and filter; any Fe_2O_3 will remain on filter, ignite it and weigh, which deduct from the impure WoO_3.

MANGANESE AND CHROMIUM

Evaporate the filtrate from the SiO_2 plus WoO_3 with 50 cc. HNO_3 to get rid of the HCl, add 175 cc. HNO_3 and precipitate the manganese by the $KClO_3$ method, using 10 grams $KClO_3$ and boiling off all Cl. Filter the MnO_2 on asbestos and wash with HNO_3. This can be used for manganese and finished as under gravimetric method for manganese in ores, iron and steel.

Cool the filtrate containing the CrO_3 and dilute to 500 cc. with cold water. The temperature should not be over 20° C. Now add a measured volume of standard solution $FeSO_4$ and titrate excess over that oxidized by the CrO_3 with standard $K_2Cr_2O_7$.

Find the amount of iron oxidized by the CrO_3, which multiplied by 0.31 equals the Cr in the steel.

Note: If no tungsten is present in the steel, the Cr may be determined direct by proceeding as in $KClO_3$ method, being careful to boil off all Cl and determine the Cr in filtrate as above.

CARBON

Do by the combustion method as given for steel and iron, except wash the carbonaceous residue with water only. HCl will give off hydrocarbons and make results low.

PHOSPHORUS

Dissolve two grams in dish with 50 cc. HNO_3 (1.20) + eight cc. NH_4OH, and adding a little HCl from time to time if it will not dissolve without, as steels containing over one per cent. Cr are not completely soluble in HNO_3. If soluble in HNO_3 only, oxidize with $KMnO_4$ and finish as in steel. If HCl is necessary, take to dryness and heat until all N_2O_4 is off and phosphorus oxidized, take up in 20 cc. HCl and again evaporate until Fe_2Cl_6 begins to separate, add 10 cc. HNO_3, heat, dilute with water, filter and precipitate by adding 50 cc. molybdic after solution has been heated to 80° C, finish as other phosphorus work.

NICKEL IN STEEL

The following method worked out by Dr. Geo. T. Dougherty and published in detail in "Iron Age" of April 25, 1907.

One gram steel is dissolved in 15 cc. HNO_3 (1.20). Have ready in a 500 cc. flask, 100 cc. HCl plus 80 cc. H_2O plus 100 cc. NH_4OH or equivalent solution of NH_4Cl in H_2O (75 grams NH_4Cl—270 H_2O). Have solution hot and add the solution of steel in HNO_3 and shake. If there is a precipitate, clear with HCl, now add NH_4OH until a dark wine color, and then add at once 50 cc. NH_4OH, shake well and pour into 500 graduated flask and dilute to the mark, mix well and filter off 250 cc. through a dry filter, add 15 cc. $HC_2H_3O_2$ and then HCl to just acid, now add NH_4OH to just alkaline, heat and pass H_2S until all the Ni is precipitated, add 15 cc. $HC_2H_3O_2$ (50 per cent.) and boil five minutes, filter and wash well with hot H_2O, ignite slowly in porcelain crucible and finally heat strongly on blast-lamp, cool, moisten with HNO_3, evaporate off HNO_3 and ignite again and weigh as NiO. Calculate to Ni.

In case Cu is present, start analysis with 20 cc. HCl (1.1) and when in solution add 2.5 cc. HNO_3. Make the 250 cc. acid with HCl and add 14 cc. (equal to five per cent. excess) HCl, pass H_2S, filter and wash with H_2S water. This filtrate is now treated as in the preceding directions and finished for Ni.

Where many analyses are to be made, the following volumetric method is best:

VOLUMETRIC METHOD

The 250 cc. are made just acid with HCl and then add NH_4OH (not more than one cc. excess). Now add five cc. $AgNO_3$ (0.1 in 200 H_2O), then five cc. two per cent. solution of KI, stir and titrate with standard KCN until last drop just destroys the opacity of the liquid.

Deduct 0.2 cc. and multiply by two and the Ni factor of the KCN.

Note: Neutralizing the steel solution too cold or making too acid or too neutral may make results 0.1 per cent. low.

STANDARD KCN SOLUTION

KCN, 24 grams to 2000 cc. H_2O, this must be standardized every two days.

For standardizing, use a standard nickel solution or a steel which has been standardized for Ni.

STANDARD NICKEL SOLUTION

Ni, Merck's "highest purity," 2.004 grams dissolved in HNO_3 and diluted to 2000 cc. with H_2O. Each cc. equals 0.001 Ni.

TO STANDARDIZE KCN

Take 50 cc. Ni solution (equal to 0.05 gram Ni), add 100 cc. H_2O, 70 cc. HCl and 70 cc. NH_4OH, make just alkaline with NH_4OH, cool, add five cc. of the $AgNO_3$ and five cc. of the KI solutions, titrate with the KCN as above, deduct 0.2 cc. and divide by 0.05; one cc. equals about 0.0025 Ni.

Note: All Ni is removed from the $Fe_2(OH)_6$ in this method by the one precipitation, and the results are just as accurate as by the well known "Ether Method."

The method also has the advantage of being cheaper and also more rapid.

TITANIUM IN ORES, IRON AND STEEL

Fuse 0.5 gram ore (finely pulverized) with eight grams of pure potassium bisulfate in platinum crucible, heat gently until the mass melts and then raise temperature until white fumes begin to come off, and keep at this temperature for 30 minutes. Do not heat too strongly or fusion will be spoiled. Cool and pour the contents and digest in not over 300 cc. cold H_2O until everything is dissolved except the SiO_2. If diluted too much, the TiO_2 may separate out. The solution may take some time, but it will do so in time. After fusing, cool and add a little H_2SO_4 and heat again to melting. This will aid in the solution. Filter off the SiO_2, dilute to 1200 cc., add Na_2SO_3 to reduce all iron, and boil six hours, replacing the water as it evaporates by some that has just been boiled. The Na_2SO_3 is five per cent. solution made distinctly acid by H_2SO_4.

The TiO_2 is now filtered off, washed by H_2O containing a little H_2SO_4, ignited, cooled and moistened by a solution of $(NH_4)_2CO_3$, ignited again and weighed. If process has been properly carried out the TiO_2 is white with a yellow tinge.

Iron and steel are done by dissolving in HNO_3 (1.20) and a little HCl, then adding 10 cc. H_2SO_4, and taking down until fumes of SO_3 come off freely, cool, dilute with H_2O, boil and filter off the SiO_2. The filtrate is diluted, reduced by Na_2SO_3 and finished for Ti as in ores.

A NEW METHOD FOR DETERMINATION OF TITANIUM

This method is based on a statement in Prescott and Johnson's work on "Qualitative Analysis," that "Sodium phosphate precipitates the titanium almost completely, even in the presence of strong hydrochloric acid." Substituting ammonium phosphate as a precipitant in order to facilitate the washing, and precipitating while boiling in about a 10 per cent. solution of hydrochloric acid, the titanium is completely thrown down as a white flocculent, somewhat gelatinous precipitate, insoluble in an excess of the precipitant; while aluminum, iron, manganese, etc., remain in solution. Only zirconium and tin interfere. Here we have at once an easy and rapid method. A determination can be done in about two hours.

The method is as follows: Dissolve from 0.3 to 0.5 gram of ferro-titanium in 15 cc. dilute nitro-sulphuric acid (equal parts nitric acid 1.20 and sulphuric acid one to three parts water) in a No. 2 Griffin beaker, evaporate to dryness or until fumes of sulfur trioxid come off, cool and add 15 cc. hydrochloric acid, 1.10 specific gravity; boil, filter and wash once with acid and about five times with warm water. To the filtrate in a No. 3 Griffin beaker add enough sulphurous acid (or ammonium bisulfite) to deoxidize the iron, and then, while boiling, 25 cc. of a 10 per cent. solution of ammonium phosphate, and continue boiling for 20 to 30 minutes; allow to settle for nearly an hour, filter and wash with hot water. The precipitate has a tendency to go through the filter, but if pulp is used, a little suction may be applied if desired. Dry and ignite, the filter with the precipitate in a tared platinum crucible at a high temperature. Cool in desiccator and weigh. The titanium factor is 0.336.

Opinions seem to be divided as to the composition of the salt. According to Roscoe and Schorlemmer, it is the basic titanium phosphate, the formula of which is $Ti_2P_2O_9$. The writer has been unable to verify this, as analysis shows more titanium and phosphorus than the above formula represents. Using the factor suggested above—viz., 0.336, corresponding to $Ti_2P_2O_9$—gives results agreeing closely with those obtained by the old methods. Ores may be fused with a mixture of potassium and sodium carbonate, transferred to a porcelain casserole and boiled with water. Allow to cool before filtering, as the titanates are not completely insoluble in water, especially in hot; filter and wash with water. Transfer filter with precipitate back to casserole, add 30 to 40 cc. hydrochloric acid, 1.10 specific gravity, boil, filter and wash with hot water into a 500-cc. flask and filtrate to mark. Pipette off a definite amount, evaporate to dryness, redissolve in 10 cc. strong hydrochloric acid and proceed as directed above. Should a very heavy precipitate be thrown down, it is necessary to pipette off a smaller sample, and conversely, a larger.

Note: The above method is by Eric John Ericson, and was given in "Iron Age" of August 27, 1903.

ANALYSIS OF WATER FOR BOILER PURPOSES

Evaporate 1000 cc. in a weighed platinum dish and heat in an oven at 100° C for one-half hour, cool in desiccator and weigh for total solids. Ignite dish at just a visible red heat for a few minutes, cool in desiccator and weigh again. The loss is organic matter, combined water and volatile matter.

Add 15 cc. HCl and 25 cc. H_2O and heat until all is in solution except the SiO_2. Be careful that all $CaSO_4$ is in solution. Filter and ignite SiO_2.

Make filtrate just alkaline with NH_4OH, boil and filter, ignite and weigh the Fe_2O_3 plus Al_2O_3.

Heat filtrate to boiling and add 10 cc. $(NH_4)_2C_2O_4$ solution, let settle, filter, ignite and weigh as CaO.

Evaporate filtrate to about 150 cc., cool, add 10 cc. $(NH_4)_2HPO_4$ solution and then 25 cc. NH_4OH, when precipitation is complete, filter and wash with water containing one-fourth its volume NH_4OH, ignite and weigh as $Mg_2P_2O_7$. Calculate to MgO.

Alkalies may be approximately determined by evaporating 200 cc. to dryness with a few drops H_2SO_4, heating gently at a low red heat until all fumes of SO_3 are off, then moisten with a solution of $(NH_4)_2CO_3$ and heating again gently to remove all acid held as bisulfate. The Ca and Mg and any alkalies are now as sulfates and are weighed as such. Calculate the CaO and MgO to sulfates, add them and the SiO_2 plus Fe_2O_3 plus Al_2O_3 together and subtract from the results by H_2SO_4. Any balance is Na_2SO_4.

Chlorin is determined in 100 cc. (or if the Cl is very low, by evaporating 300 cc. to 100 cc.), by a standard solution of $AgNO_3$, using a few drops of K_2CrO_4 solution, the presence of any excess $AgNO_3$ will form the red silver chromate in the solution. It is best in order to get accurate results to run a blank with distilled water, using same amount of K_2CrO_4 and adding $AgNO_3$ until colors match, and then deduct the blank from the other results.

If there is much Cl in sample to be analyzed, it is best to add nearly a like amount of standard NaCl to the blank and then subtract only the $AgNO_3$ used in excess of that required by the NaCl. If water is acid, add a little Na_2CO_3 solution.

For SO_3, evaporate 500 cc. to about 150 cc. and make acid with HCl. While boiling, add 10 cc. $BaCl_2$ solution, let stand several hours, filter, ignite, and weigh the $BaSO_4$. Calculate to SO_3.

Note: The amount of water used can be varied somewhat according to whether it carries a large or small amount of substance to be determined.

The SiO_2 is usually in so small an amount that the sample can be evaporated to about 150 cc., the HCl added and the SiO_2 and Al_2O_3 and Fe_2O_3 all precipitated together by NH_4OH, and reported in the same manner. In this case a separate sample is run for total solids. The filtrate from the SiO_2 and Al_2O_3 and Fe_2O_3 may be divided and a portion finished for SO_3.

SOLUTIONS FOR Cl DETERMINATION, SODIUM CHLORID

Heat some of the c. p. salt over a bunsen burner not quite to redness for a few minutes, cool and dissolve 0.824 gram in 500 cc. H_2O, one cc.=0.001 gram Cl.

SILVER NITRATE

Five grams $AgNO_3$ in 1000 cc. H_2O, determine its value by titrating with NaCl solution.

POTASSIUM CHROMATE

Five-tenths gram K_2CrO_4 in 100 cc. H_2O, add $AgNO_3$ solution until a red precipitate is produced, filter and use the clear solution.

CALCULATION OF RESULTS AND REPORTING SAME

Combine the alkalies with the Cl and any excess with the SO_3. Should there be more Cl than will combine with the alkalies, combine it with Ca and then with the Mg. Should there be more than enough alkalies to combine with both the Cl and SO_3, calculate the balance to carbonates. Any SO_3 left after the alkalies are saturated is combined with the Ca and then the Mg. Any Ca and Mg left after the Cl and SO_3 are saturated is calculated as carbonates.

Milligrams per 1000 cc. = parts per million. Report results in parts per million and grains per U. S. gallon.

DETERMINATION OF TALLOW FAT IN CYLINDER OILS

Weigh 10 grams into a No. 2 beaker, add 50 cc. ethyl alcohol (95 per cent.) in which is dissolved about four grams NaOH, heat to not above 140° C until all the alcohol is driven off, cool, add 75 cc. hot water and digest until all soap is in solution, wash into a separatory funnel, cool and add 75 cc. ether (alcohol free), shake well for several minutes and let stand until the ether and water solutions have completely separated, which may take several hours.

Draw off the water solution of the soap representing the tallow fat, heat to drive off all ether, make acid by adding H_2SO_4, and add at once a weighed amount (about five grams) of white wax, let stand at near the boiling point until all fatty acids have separated from the water and have been absorbed by the wax, cool, remove the wax and wash it and the beaker with H_2O, dry beaker, which has a little wax adhering to it, by heating on steam plate, cool and weigh; now remove wax and weigh again to get weight of wax adhering to it.

Dry the thin cake of wax with blotting paper and finally over $CaCl_2$ and weigh. The difference in the weights before and after absorbing is the fatty acids, which multiplied by 0.97 equals the tallow fats, or more correctly, all other fats than mineral. Cylinder oils should contain from five per cent. to seven per cent. tallow fat.

GAS ANALYSIS

BLAST-FURNACE AND CHIMNEY GASES

SAMPLING

Glass or porcelain tubes can be used in all cases except where the heat is so high as to melt them. On account of their liability to break, however, they are not to be recommended. A water jacketed tube is best. Make same according to the directions in "Gas and Fuel Analysis," Gill. Plain metal tubes should never be used in taking samples for analysis except at low temperatures, as the composition of the gas may be materially changed.

Blast-furnace gases which are under pressure do not need to be aspirated, but for chimney gases it is necessary. A steam or air pump is the best arrangement for this purpose. The author has used the Richards jet pump with very good results. By the use of a tee tube or by aspirating through a large bottle, the gas is permitted to flow continuously and the sample may be taken from the tee or the bottle and will represent the gas for a period of several minutes, or by filling the sampling tubes very slowly a sample may be taken covering a longer period of time. Sampling tubes are made of timed iron and are made small at each end for the attachment of a piece of rubber tubing and pinch-cock. The tubes should hold 200 or 300 cc. In taking the sample, the tube should be filled with water which is saturated with NaCl and also with the gas to be sampled. This water is also used in the apparatus for analyzing the gas. By allowing this solution to run from the tube, the gas is drawn in, and when filled the pinch-cocks are closed and taken to laboratory for analysis. Samples should be analyzed at once or results will not be reliable.

METHOD OF ANALYSIS

The Orstat apparatus made with four absorption pipettes is all that is to be desired for this work. Fill the first pipette with the NaOH solution, the second with the pyrogallate and the third and fourth with CuCl. Fill small aspirator bottle with the water saturated with NaCl and the gas, to which also is added a few drops H_2SO_4, bring to the top of the measuring burette and close stop-cock, now attach the tube filled with gas, having other end dipping under some of the NaCl solution, open pinch-cocks, lower the bottle, turn stop-cock and draw in about 25 cc. gas, turn the three-way stop-cock, and by raising the bottle expel all the gas again from the apparatus. This will carry with it any air that was in the tubes. Now by lowering the bottle, draw in 100 cc. gas and close the stop-cock. Now, having solutions in all pipettes at the 0-mark, pass the gas into the NaOH solution three times and then measure; the loss in cc.=per cent. CO_2. Next pass into the pyrogallate until there is no further loss in volume; this loss is O. Next pass into the first CuCl pipette until most of the CO is removed, then pass several times into the other CuCl pipette, which will quickly remove the last of the CO. The gas not absorbed is counted as N.

DUST DETERMINATION

APPARATUS

This apparatus is made of two concave pieces of brass with a ring extension to allow of clamping together by means of bolts and thumb nuts. This forms a small chamber and an air tight joint is made by inserting a rubber gasket. A wire gauze is also placed between the two cups to support a weighed filter paper. Each cup has attached to its outer end a one-quarter-inch brass tubing, one of them of sufficient length to pass well within the inner wall of the gas chamber. This tube also carries on its end next the cup a one-half-inch plug, threaded to fit the opening in the gas chamber. The tube leading from the other cup is connected by means of a piece of rubber tubing, with a one-quarter-inch pipe which leads to a convenient place for the measuring of the gas taken.

An oil barrel placed on end and filled with water is a convenient method of measuring the gas and is operated as follows: Have a valve attached near bottom of barrel for drawing off the water, and at the top end a rubber stopper with three holes. One of these is connected with the pipe leading to the gas chamber by means of rubber tubing and pinch-cock. Another is connected to a water gauge to show the pressure or vacuum, and the third is for a thermometer.

METHOD OF DETERMINATION

Have barrel filled with water and all connections made at that end and tubing closed by pinch-cocks. Now attach the dust cup to the hose at the other end of pipe, remove plug from hole in gas chamber and insert tube, now open pinch-cocks at barrel and note gas pressure, now draw off water quite rapidly until you have taken from 100 to 300 pounds (from which the cubic feet of gas are calculated), close pinch-cock on tube carrying gas, which, if done properly, the gas pressure in barrel can be brought to the atmospheric pressure. Note the final pressure and temperature of gas in barrel and also the pounds of water taken, from which calculate the grains dust per cubic foot at a standard pressure and temperature. Remove dust cups and dry filter paper and weigh to determine amount of dust.

Note: When the dust in the gas is being removed by a system of washing, two such forms of apparatus may be operated at the same time in order to determine the percentage of dust removed.

In removing the gas and dust from the chamber, the tube should be inserted at a right angle to the vertical side of the same.

For extreme accuracy it is also necessary to take the barometric pressure for use in making calculations.

MOISTURE DETERMINATION APPARATUS

For measuring, taking temperature and pressure, the same apparatus is used as in dust determination. Two may also be run at the same time to determine moisture both before and after washing. For the absorption of the moisture, use the following train:

A small $CaCl_2$ tube is filled with cotton and dried. The small end is inserted in a rubber stopper to fit hole in gas chamber. (A cork stopper is used if heat is strong enough to melt the rubber). To the other end of the tube is connected a large tube filled with fused $CaCl_2$, and this is next connected with a Mohr bulb containing H_2SO_4. This is connected with the pipe leading to the measuring apparatus.

METHOD OF DETERMINATION

The tubes are all weighed and connected.

The gas is now aspirated same as in the dust determination, only much more slowly, and not more than one or two cubic feet taken. Note pressure and temperature, detach apparatus and weigh, remove cotton from tube, which also contains some dust, dry it and tube, and weigh again. Take the sum of the amount of water found in the tubes and calculate to grains per cubic foot at standard temperature and pressure.

Note: If gas is hot enough to burn cotton in tubes, fill that portion of the tube nearest gas chamber with glass beads.

If H is to be determined, do same as given under Producer and Coke-Oven Gases.

PRODUCER AND COKE-OVEN GASES

SAMPLING

Take samples by same method as given under Blast-Furnace and Chimney Gases.

APPARATUS

For all practical purposes the Orstat apparatus may be used, together with a palladium tube for burning the H and a CuO tube for burning the CH_4. If C_2H_4 is to be determined separately, it is done after removing CO_2, by passing into a pipette containing fuming $H_2S_2O_8$, and again into the KOH to remove any acid fumes. As the C_2H_4 is so small it is as well determined along with the CO and counted as such. The Pd tube is connected with the three-way stop-cock and the bulb of the same placed in a dish resting on a wire gauze with gas burner underneath. The other end is connected to a one-quarter-inch brass tube about nine inches long containing CuO and placed over a blast-lamp. The other end of the brass tube is attached to a simple two-bulb pipette, having a stop-cock between the bulbs. The pipette contains H_2O or Hg to act as a seal. Hg is best, as it will not absorb the gas.

METHOD OF ANALYSIS

Proceed exactly as in analysis of Blast-Furnace Gases until CO is removed. Now disconnect the tube which contained the sample of gas for analysis, attach a rubber tube leading to an O tank, open stop-cock and run in O until there is again about 100 cc. gas in apparatus. Close stop-cock, bring Hg into capillary of pipette and close stop-cock and make all connections. Now open stop-cock leading to Pd tube and take reading in measuring tube, fill dish with hot H_2O, light burner and bring to boiling, elevate aspirator bottle, open stop-cock in Hg pipette and pass gas several times. This will burn all the H forming H_2O and any CO, which has not been absorbed by the CuCl, to CO_2. Bring Hg into capillary and close stop-cock of pipette, fill dish with H_2O at room temperature and let stand a few minutes, take reading, which will show contraction due to H and any CO, pass into KOH. Any CO_2 found is equal to CO not absorbed by the CuCl. Now heat brass tube to redness, keeping ends cool by cotton saturated with H_2O, pass gas in same manner as for H and bring Hg into capillary and close stop-cock, remove blast-lamp and cool brass tube with H_2O, lower aspirator bottle and disconnect carefully where attached to Hg, pipette and allow air to flow through the brass tube until there is again about 100 cc. in apparatus, close three-way stop-cock and let stand a few minutes. By drawing this much air into apparatus all CO_2 in CuO and Pd tubes is taken with it. Note the reading, pass into KOH and read again. Any CO_2 is directly the amount of CH_4 in the gas.

CALCULATION OF H + CO AND OF CH_4

As two volumes of CO unite with one volume of O to form two volumes of CO_2, the CO_2 found after burning in Pd tube is directly the amount of CO, and must be added to the CO found by CuCl.

One-half the CO is equal to the contraction caused by the burning of the CO to CO_2, therefore, one-half the CO found along with the H, subtracted from the total contraction caused by the H plus CO, equals the contraction caused by the H.

As two volumes of H unite with one volume of O to form H_2O, two-thirds the contraction caused by H equals H.

As the C in one volume of CH_4 unites with one volume of O to form CO_2, the final CO_2 found is directly the amount of CH_4. Add together the different gases found and subtract from 100 to find the N.

Note: In burning the CH_4, there may be an absorption of O due to the oxidation of the brass tube; but this is unimportant, however, as it is only the amount of CO_2 formed that need be determined.

The above method is about the same as given by

Blair, except that the Pd tube is heated by boiling H_2O instead of by a direct flame, thereby doing away with any danger of heating too highly and burning CH_4. Also the burning of the CH_4 is done in a small brass tube instead of in a combustion furnace, and the CO_2 formed is absorbed in the KOH pipette and measured instead of passing into $Ba(OH)_2$ and titrating, thereby making the method more simple and also shortening the time very materially.

A fresh solution of CuCl will absorb practically all the CO, but when it becomes partially saturated, it will not do so. A gas not containing any CO, if passed into a solution of CuCl which is nearly saturated by CO, will take up CO from the CuCl.

BEARING METALS

ALLOYS CONTAINING TIN, ANTIMONY, COPPER AND LEAD

Treat one gram of the finely divided alloy with 25 cc. HNO_3 and evaporate to a paste, add 10 cc. HNO_3 (1.20) and dilute with hot water to about 50 cc., filter and wash with water containing about five per cent. HNO_3, ignite and weigh as SnO_2 plus Sb_2O_4, which usually contains about one-tenth per cent. Cu. To filtrate, add five cc. H_2SO_4 and evaporate to fuming, cool, dilute with 100 cc. H_2O, boil, filter and wash with H_2O containing a little H_2SO_4. Burn slowly in porcelain crucible on argand burner and weigh as $PbSO_4$. Calculate to Pb. Evaporate the filtrate to about 50 cc., cool, add NH_4OH until alkaline, then $HC_2H_3O_2$ about two cc. in excess, cool again, add from one to three grams KI, let stand a few minutes and titrate with $\frac{N}{10}$ $Na_2S_2O_3$ which has been standardized by pure Cu. Use freshly prepared starch solution as indicator. Iron is the only element that is likely to be present that will interfere, and if present must be filtered off after adding the NH_4OH.

For Sn, dissolve one-half gram in an erlenmeyer flask by treating with 10 cc. HCl, and after heating a few minutes add a little $KClO_3$, boil nearly to dryness to expel all Cl, add 30 cc. HCl and 70 cc. H_2O. Now add to flask a coil of pure iron wire, connect flask by means of a rubber stopper with a tube, the end of which dips into a strong solution of $NaHCO_3$, heat and boil slowly until all Sn and Sb are reduced (the Sb to the metallic state as a black precipitate) and all the Fe is dissolved, now cool while keeping tube in the $NaHCO_3$. When cold, add starch solution and titrate with $\frac{N}{10}$ iodin. One cc. iodin = 0.0059 gram Sn. Calculate to SnO_2, subtract from total SnO_2 plus Sb_2O_4, and then calculate the Sb_2O_4 to Sb. The $\frac{N}{10}$ iodin may be standardized by pure Sn or by $\frac{N}{10}$ $Na_2S_2O_3$.

Note: Small amounts of Cu do not interfere with the titration of Sn by N/10 iodin if no local excess of iodin is allowed. For alloys low in Sn and high in Sb, add more Sn or all Sb will not be removed as Sb_2O_4.

For alloys high in Cu, the following method is used:

Method for alloys containing Sn, Sb and Pb and which are high in Cu.

Dissolve one gram in 20 cc. HCl with little $KClO_3$, dilute with 15 cc. H_2O, and if much $PbCl_2$ separates, filter it off and save for the Pb determination. Add to the solution two to four grams $H_2C_4H_4O_6$, neutralize most of the acid with NaOH, and then add to a solution of 15 grams NaOH in about 200 cc. H_2O, pass H_2S until all Pb and Cu plus any Fe and Bi are precipitated and a small amount of Na_2S is formed, filter and wash with water containing a little Na_2S. The filtrate containing the Sn and Sb is divided into two equal portions, an excess of HCl added and boiled to expel all H_2S. A little $KClO_3$ is now added and boiled until all is in solution except a little S and all Cl is expelled. Now reduce the Sn with iron wire, as in other method, and titrate with $\frac{N}{10}$ iodin. The two portions should check closely. Do the combined SnO_2 plus Sb_2O_4 on a separate sample and calculate the Sn and Sb as in the other method.

Dissolve the sulfids of Cu and Pb and any Fe, etc., in HNO_3 (1.20) or use the filtrate from the combined oxids and determine the Pb as $PbSO_4$ and the Cu iodometrically as in the previous method. Any Fe and Bi can be removed by NH_4OH and $(NH_4)_2CO_3$. The latter are present only as impurities and are not determined if in small amounts. The Fe may come from the NaOH used.

ALLOYS CONTAINING COPPER, TIN AND ZINC

Dissolve two grams of the finely divided alloy in HNO_3 and evaporate nearly to dryness, add 10 cc. HNO_3 (1.20) and dilute to 50 cc. with hot H_2O, filter, ignite and weigh with impure SnO_2, mix with 10 times its weight of $Na_2S_2O_3$ and fuse, but do not heat too strongly; dissolve in hot H_2O and filter off any sulfids of CuPb and Fe, ignite and weigh, which subtracted from the first weight gives closely the weight of SnO_2. The Sn is more accurately determined by adding HCl to the filtrate from the sulfids, reducing with iron wire and titrating with $\frac{N}{10}$ iodin, as in alloys containing Sn, Sb, Cu, and Pb.

The sulfids of Cu, Pb, etc., are dissolved in HNO_3 (1.20) and added to the filtrate from the impure SnO_2, 10 cc. H_2SO_4 are added and evaporated to fumes, cooled and diluted to about 100 cc. with hot H_2O, boiled, filtered and washed with H_2O containing a little H_2SO_4, ignited in porcelain crucible on argand burner and weighed as $PbSO_4$. Calculate to Pb.

The filtrate from the Pb is diluted to 200 cc. and mixed. Determine Cu in 50 cc. as follows: Make alkaline with NH_4OH and filter off any $Fe_2(OH)_6$ (which, if an appreciable amount, is ignited and weighed), add $HC_2H_3O_2$ two cc. in excess, cool, add three grams KI, let stand several minutes and titrate with $\frac{N}{10}$ $Na_2S_2O_3$ which has been standardized by pure Cu.

To the remaining 150 cc. warm solution, add H_2SO_3 in excess and then ammonium thiocyanate to remove the Cu and let stand until it settles, clear, filter and wash with H_2O, boil to expel H_2SO_3, pass H_2S and filter off any Sb, As, Bi, etc., boil again to expel H_2S, cool somewhat and oxidize by adding HNO_3, being careful not to inhale the HCN, make alkaline with NH_4OH and filter off any $Fe_2(OH)_6$. If the above elements are not present in appreciable amounts, the Zn can be determined without removing them or the excess thiocyanate.

To the acid or alkaline solution, add 10 cc. HCl and make faintly alkaline with NH_4OH and then faintly acid with HCl, heat nearly to boiling and add 20 times the weight of $(NH_4)_2$-

HPO_4 that there is Zn present, heat until the precipitate becomes crystalline, filter, washing out beaker with the mother liquor, wash with hot H_2O, ignite in porcelain and weigh as $Zn_2P_2O_7$. Calculate to Zn.

Note: The above methods, will serve for the analysis of most bearing metals. For a complete treatise on methods for analysis of these alloys, consult "Analysis of Steel Works Materials," Brearley and Ibbotson.

ALLOYS CONTAINING PHOSPHORUS

Decompose 0.5 gram alloy with HNO_3 and evaporate nearly to dryness, add 10 cc. HNO_3 (1.20), dilute with H_2O and filter, ignite in porcelain crucible and fuse in same at a red heat with KCN, extract with H_2O and filter, make filtrate acid with HCl and boil off HCN. If phosphorus is very high, it is best to precipitate it in this solution with magnesia mixture and weigh as $Mg_2P_2O_7$. However, if as low as one per cent., it is more rapid to do as molybdate, as follows: Add a little Fe_2Cl_6 and a few drops HNO_3 to the solution, make alkaline with NH_4OH and filter off the $Fe_2(OH)_6$ which contains all the phosphorus, dissolve in a little HNO_3 (1.20), precipitate by adding molybdic and finish in the usual way.

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